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**QUATERNARY AND HIGHER ORDER ALLOY  
ADDITIONS TO OXIDE DISPERSION  
STRENGTHENED HIGH VOLUME FRACTION  
GAMMA PRIME Ni-Cr-Al ALLOYS MADE  
BY MECHANICAL ALLOYING**

**BY  
R.C. BENN**

**DECEMBER 1977  
FINAL REPORT**

**"APPROVED FOR PUBLIC RELEASE - DISTRIBUTION UNLIMITED"**

**PREPARED UNDER CONTRACT N62269-76-C-0483**

**NAVAL AIR DEVELOPMENT CENTER**

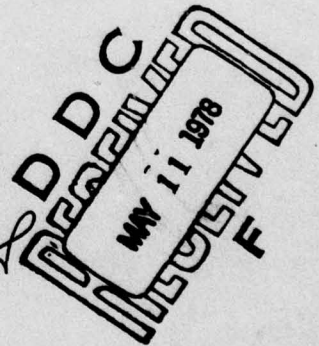
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**FOR**

**NAVAL AIR SYSTEMS COMMAND**

**DEPARTMENT OF THE NAVY**

**WASHINGTON, D.C. 20361**



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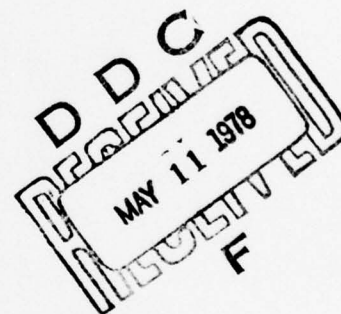
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PRIME Ni-Cr-Al ALLOYS MADE BY MECHANICAL ALLOYING

Final Report  
Contract N62269-76-C-0483  
December, 1977

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The object of this work was to develop ODS alloys which would derive a significant high temperature strength increment from the reten- tion of high volume fractions (>50%) of $\gamma'$ at 2000°F (1095°C). This investigation has identified the nature and level of quaternary addition elements that are beneficial to the directional recrystal- lization response and properties of ODS, high volume fraction $\gamma'$ , Ni-Cr-Al alloys made by mechanical alloying. Specifically, the effects of quaternary additions of W, Ta, Nb, Mo, Co, Hf and Ti were investigated. In particular, the quaternary alloys with W and Mo		

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respectively exhibited significant high temperature strength advantages over DS Mar M-200 + Hf and a current DS  $\gamma/\gamma'$ - $\alpha$  eutectic. Raising the Cr level in the ternary Ni-Cr-Al base alloy significantly improved the corrosion resistance. Oxidation resistance of the experimental alloys was excellent. The data from the simple ternary and quaternary alloys was used to initiate a program of designing more complex alloys.

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## TABLE OF CONTENTS

	<u>Page No.</u>
I. INTRODUCTION . . . . .	1
II. EXPERIMENTAL PROCEDURES . . . . .	2
2.1 Attritor Processing . . . . .	2
2.2 Extrusion . . . . .	2
2.3 Heat Treatment . . . . .	3
2.4 Mechanical Testing . . . . .	4
2.5 Physical and Chemical Testing . . . . .	4
III. TECHNICAL PROGRESS SUMMARY . . . . .	5
3.1 Evaluation of Series I Alloys . . . . .	5
3.1.1 Powder Processing of Series I Alloys.	5
3.1.2 Thermomechanical Processing . . . . .	5
3.1.3 Gradient Annealing Studies . . . . .	6
3.1.3.1 Effect of Tungsten . . . . .	6
3.1.3.2 Effect of Tantalum . . . . .	6
3.1.3.3 Effect of Niobium . . . . .	6
3.1.3.4 Effect of Molybdenum . . . . .	6
3.1.3.5 Effect of Cobalt . . . . .	6
3.1.3.6 Effect of Hafnium . . . . .	7
3.1.3.7 Effect of Titanium . . . . .	7
3.1.3.8 Effect of Chromium . . . . .	7
3.1.4 Zone Annealing . . . . .	8
3.1.5 Mechanical Property Evaluation . . . . .	8
3.1.5.1 Stress Rupture . . . . .	8
3.1.5.2 Tensile Test . . . . .	9
3.1.6 Gamma Prime Volume Fraction . . . . .	9
3.1.7 Hot Corrosion Evaluation . . . . .	9
3.2 Evaluation of Series II Alloys . . . . .	10
3.2.1 Powder Processing of Series II Alloys.	10
3.2.2 Thermomechanical Processing . . . . .	10
3.2.3 Gradient Annealing Studies . . . . .	10
3.3 General Discussion . . . . .	11
IV. SUMMARY . . . . .	14
ACKNOWLEDGEMENT . . . . .	14
REFERENCES . . . . .	15
TABLES I-XV	
FIGURES 1-42	

FINAL REPORT  
QUATERNARY AND HIGHER ORDER ALLOY ADDITIONS TO  
OXIDE DISPERSION STRENGTHENED HIGH VOLUME  
FRACTION GAMMA PRIME Ni-Cr-Al ALLOYS  
MADE BY MECHANICAL ALLOYING

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I. INTRODUCTION

Today, several distinct types of alloys with directional structures offer potential for increased operating temperatures (100-350°F) for gas turbine vanes and blades. Among these are the oxide dispersion strengthened (ODS) alloys(1-3) produced by mechanical alloying(4). The ODS +  $\gamma'$  nickel-base superalloys which have been produced to date offer substantial increases in high temperature strength capability over conventional nickel-base superalloys.

In these previous ODS alloy investigations, alloys with conventional volume fractions of  $\gamma'$  (30-50%), and low  $\gamma'$  solvus temperatures were studied. At intermediate temperatures (1400°F[760°C]) the ODS +  $\gamma'$  alloys enjoy a significant strength increment over their  $\gamma'$ -free counterparts; e.g., TD-Ni and TD-Ni-Cr. As the use temperatures are raised, the  $\gamma'$  solvus is approached and the  $\gamma'$  strengthening increment decreases to zero. This occurs at approximately 1600-1800°F (870-980°C) for present ODS +  $\gamma'$  alloys.

Work was initiated under Contract N00019-75-C-0313 to develop an ODS +  $\gamma'$  alloy with a very high volume fraction of  $\gamma'$  at the intended use temperature of 2000°F (1095°C). The intention of that program was to combine  $\gamma'$  and ODS strengthening at 2000°F (1095°C). The results of this previous work(5) show that it is possible to produce an oxide dispersion strengthened high volume fraction  $\gamma'$  (90%) Ni-Cr-Al alloy, designated alloy 2, with the coarse elongated grain structures necessary for good high temperature strength.

Through quaternary alloy additions of tungsten to the alloy 2 base composition (Ni-10 wt.%Cr-9 wt.%Al-1.1 wt.%  $Y_2O_3$ ), further improvements in high temperature strength were achieved. Despite a small increase in density, the highest tungsten alloy 8 (~7 wt.%W) showed superior 1000 hour density corrected specific rupture strength, when compared to the DS Mar M-200 (above 1530°F [830°C]) and a current DS  $\gamma/\gamma'-\alpha$  eutectic alloy (above 1630°F [890°C]).

An investigation of quaternary titanium additions to the alloy 2 base composition showed that this element appeared to be detrimental to thermochemical processing and recrystallization response. At a level of 2.8 wt.%, only limited recrystallization was observed. Therefore, except in small quantities (<2.5 wt.%), titanium cannot be used as a  $\gamma'$  strengthener in these alloys.

Based on the feasibility and modest improvements demonstrated under the previous contract, a new program was initiated to conduct follow-on research to continue alloy development of high volume %  $\gamma'$  MA nickel-base superalloys. This effort was conducted under NAVAIR Contract N62269-76-C-0483.

The objective of this program was to improve the mechanical and chemical properties of the high volume %  $\gamma'$  Ni-Cr-Al ODS alloy through quaternary and higher order alloy additions.

This is the final report for Contract N62269-76-C-0483.

## II. EXPERIMENTAL PROCEDURES

### 2.1 Attritor Processing

The following powders were used for mechanical alloying:

- nickel powder type 123
- elemental chromium, tungsten, cobalt,  
molybdenum, tantalum and niobium
- Ni-47Al master alloy
- Ni-28Ti-17Al master alloy
- Ni-47Hf-10Al master alloy
- Y<sub>2</sub>O<sub>3</sub> (calcined yttrium oxalate)

Powder batches of the selected compositions were mechanically alloyed in the attrition mills under controlled conditions. The resultant mechanically alloyed powder was characterized using chemical analysis for O, N, C, and Fe, screen analysis, and metallographic examination. The criteria for accepting powder as well processed are outlined in Reference 4. Essentially, a powder is considered processed when metallographic examination indicates that it is completely homogeneous (e.g., Figure 1). Experience has taught that a coarse powder size distribution, normal oxygen (~0.5-0.8 wt.%) and iron (0.5-0.8 wt.%) levels are indicative of well processed powder.

### 2.2 Extrusion

After screening to remove the coarse +12 mesh particles, powder batches of each composition were cone blended for two hours and packed into 3.5 inch O.D. steel extrusion cans. Between six and nine cans were prepared for each of the compositions investigated in this program. The extrusion cans were sealed in air prior to extrusion.



Extrusions were made after preheating the billets two hours to temperatures ranging from 1850°F (1010°C) to 2150°F (1175°C). Round extrusion dies were used yielding ratios ranging from 18 to 30:1. Conical dies having an included angle of 90° were employed. Lubrication was provided by a glass pad on the die face with oil in the extrusion chamber and a glass wrap on the billet.

All extrusions were performed on a 750 ton Loewy Hydropress at throttle settings of 35 or 100%. Ram speed and pressure were continuously recorded during each extrusion. Occasional recorder malfunction resulted in no record for a few extrusions made in this work.

### 2.3 Heat Treatment

The objective of the extrusion studies was to determine the conditions required to yield a coarse elongated grain structure, in each alloy, upon heat treatment. The necessity of obtaining this structure to achieve maximum high temperature strength in ODS alloys is well documented (6,7).

The nature of the recrystallization response is generally described in terms of transverse grain diameter (fine .1-1  $\mu\text{m}$ , medium 1-50  $\mu\text{m}$  and coarse 50-250  $\mu\text{m}$ ) and grain aspect ratio (length/diameter). For optimum high temperature strength, a coarse grain size with a grain aspect ratio (GAR) of about 10 is required.

Stationary gradient anneals were used to determine the recrystallization behavior as a function of annealing temperature. Four inch long extruded bar samples were individually annealed in a gradient furnace having the thermal profile shown in Figure 2. Total annealing time was 1/2 hour. After heat treatment, these bars were surface ground parallel to the extrusion direction to reveal the recrystallized grain structure as a function of position along the gradient. This is a simple method for determining the temperature range over which recrystallization to a coarse grain structure will take place. It also pinpoints the critical heat treatment temperature at which the best coarse elongated grain structure is achieved.

Zone annealing can be an effective way of increasing the grain aspect ratio, and hence the high temperature strength, of ODS superalloys(1,8). Generally, extruded bar which shows a coarse grain structure (elongated or equiaxed) on isothermal or static gradient annealing, will respond favorably to zone annealing. Selected bars were zone annealed in the same gradient furnace (Figure 2) at between 2.7 iph (6.8 cmph) and 5.4 iph (13.6 cmph) and maximum zone temperatures ranging from 2225°F (1220°C) to 2420°F (1330°C). Specimens were cut from these zone annealed bars and sectioned to reveal the grain aspect ratio achieved.

A heat treatment study was conducted to determine the volume %  $\gamma'$  in the alloys.

#### 2.4 Mechanical Testing

Specimens for mechanical testing were ground from round heat treated bars with their tensile axis orientated parallel to the extrusion direction. In all cases this corresponded to the direction of structural elongation. Stress rupture tests were performed at 1400°F (760°C), 2000°F (1095°C), and 2100°F (1150°C). Initially, 1000/2 ksi (13.8 MPa) step loading was used to determine the creep capability of the material. Constant load tests at the above temperatures were used to determine the stress/temperature/life capability of each alloy material with the best grain structure achieved in this program. These tests were performed in accordance with the appropriate ASTM specification on specimens with .125 inch (3.18 mm) gauge diameter, gauge length of 1 inch (25.4 mm), and .25 inch (6.35 mm) -20NC threaded ends. Elongation and reduction of area were measured from the fractured specimens.

Tensile testing on selected alloy bars was performed at room temperature and 1400°F (760°C) in air. Again the test specimens were identical in dimension, orientation and method of manufacture to those used for stress rupture testing.

#### 2.5 Physical and Chemical Testing

Density measurements were made by simple calculations based on the dimensions and weight of cylindrical oxidation test specimens. This method is accurate to within 1%.

Oxidation tests were performed at 2000°F (1095°C) for 504 hours. The test was cyclic in nature with the specimens being cooled rapidly to room temperature and weighed daily. The environment was low velocity air + 5% H<sub>2</sub>O. After final weight measurements, the samples were descaled by a light Al<sub>2</sub>O<sub>3</sub> grit blast and final weight loss was measured.

Burner rig sulfidation tests were conducted at 1700°F (930°C) for 168 hours. The rig used corresponds to the G.E. Lynn low velocity burner rig(9). This test ran on a one hour cycle, 58 minutes rotating in the flame, two minutes in air blast. The flame conditions were a 30:1 air + 5 ppm seawater (ASTM Spec. D1141-52) to fuel (.3% sulfur JP-5) ratio at low velocity. The specimens were weighed each 24 hours and standard diametric (cross section) metal loss and grain boundary penetration measurements were made at the end of the test. Descaled weight loss was also determined.

A [100] texture in directional structures is known to provide a definite advantage in thermal fatigue resistance. A low elastic modulus was observed on one alloy 2 extruded and zone annealed bar. X-ray diffraction texture analysis was used to determine preferred orientations of the directional structure observed in alloy 2 bars with high and low elastic moduli.

### III. TECHNICAL PROGRESS SUMMARY

This alloy development program was conducted in two series. Series I involved quaternary additions of Ta, Nb, Hf, Ti, Mo, W and Co to the Ni-10 at.%Cr-17.5 at.%Al base composition developed earlier. The effect of these additions on the mechanical alloying and recrystallization response of extruded bar has been studied. In addition, higher chromium levels, 15 and 20 at.%, were studied in order to achieve improved sulfidation resistance. The structural evaluation of Series I alloys is complete. Those alloys showing adequate directional grain structures upon zone annealing have now been evaluated by mechanical and corrosion tests.

Series II involved quinary and higher order alloy additions. These alloys have been based on the results obtained from Series I (see Section 3.2). Several complex Series II alloys have been evaluated.

#### 3.1 Evaluation of Series I Alloys

##### 3.1.1 Powder Processing of Series I Alloys.

Powder processing of Series I alloys has been completed. A list of alloys prepared by mechanical alloying is given in Table I. Two 18.7 pound (8.5 kg) powder batches of each composition were produced. Prior to a change in composition, one wash heat was made and discarded. Each powder batch was characterized by chemical and screen analysis and by metallographic examination. The results of these powder characterization studies are given in Table II. Figure 1 shows a typical etched microstructure of one of the powder batches (alloy 9).

3.1.2 Thermomechanical Processing. After each powder batch had been qualified as well processed using established criteria(4), the two batches of each composition were mixed by cone blending and packed into mild steel extrusion cans. A minimum of four cans was prepared for each composition listed in Table I. The extrusion conditions employed for consolidation of powder are detailed in Table III. Following extrusion, the alloys were tested for recrystallization response by gradient annealing. The gradient furnace and the thermal profile utilized for both gradient annealing and zone annealing are shown in Figure 2.



3.1.3 Gradient Annealing Studies. The effects of quaternary elemental additions to the simple Ni-10 at./ 9.8 wt.%Cr-17.5 at./8.9 Wt.%Al ternary composition (Alloy 2 - see Figure 3) with respect to directional recrystallization response have been evaluated as follows:

3.1.3.1 Effect of Tungsten. Tungsten, at a level of 2 at.% (6.6 Wt.%) was evaluated under the previous contract(5). Excellent directionally aligned grains were developed (Figure 4). In the present contract, tungsten at a level of 4 at.% (12.7 wt.%) was evaluated. Figure 5 shows that the three bars extruded at 100% press throttle displayed no recrystallization response on gradient annealing. Subsequent repetition of the 2150°F (1175°C) and 2050°F (1120°C) extrusions, but at 35% throttle, initiated some recrystallization response (bar V75D in Figure 5) on gradient annealing but to no significant degree.

3.1.3.2 Effect of Tantalum. Tantalum additions were made at levels of 1, 3, and 6 at.% (3.3, 9.4, and 17.4 wt.%). Extrusion was performed using 100% press throttle at temperatures ranging from 1950°F (1065°C) to 2200°F (1205°C) using a reduction of 18:1. Suitably directionally aligned grains were obtained only for extruded bar containing 1 at.%Ta (3.3 wt.%). As tantalum is increased, the recrystallization response decreases until at the highest level, 6 at.% (17.4 wt.%), no recrystallization occurs (Figures 6-8).

3.1.3.3 Effect of Niobium. Niobium additions were made at levels of 1, 3 and 6 at.% (1.7, 5.1, and 9.8 wt.%) and also extruded 18:1 at temperatures of 1950°F (1065°C) to 2200°F (1205°C). A good elongated grain structure was obtained for the 1 at.%Nb (1.7 wt.%) alloy extruded at 2050°F (1120°C), as shown in Figure 9. No recrystallization response was obtained in the two other niobium alloys for any of the extrusion conditions examined. Macrographs for several conditions are shown in Figures 10 and 11. Note the evidence of melting at the higher annealing temperatures for these alloys, as indicated by the increase in bar diameter.

3.1.3.4 Effect of Molybdenum. Extruded bar of alloys containing 1 and 2 at.%Mo (1.8 and 3.6 wt.%Mo) gave excellent directionally aligned grains upon gradient annealing. Extrusion details are given in Table III (Alloys 15 and 16), and examples of microstructures are shown in Figures 12 and 13. Note that good elongated grains were obtained for a wide range of extrusion temperatures.

3.1.3.5 Effect of Cobalt. Cobalt additions were evaluated at levels of 5 and 10 at.% (5.5 and 11.1 wt.%). The somewhat varied recrystallization response that was obtained in bars extruded at 100% press throttle was overcome by extrusion at a slower ram speed (i.e., 35% press



throttle). Figures 14 and 15 show that bar extruded at 2100°F (1150°C) and 35% throttle gave an acceptable and similar structural response, but at a lower annealing temperature compared to material extruded at 100% throttle.

3.1.3.6 Effect of Hafnium. Hafnium has been evaluated at levels of 1 and 2 at.% (3.3 and 6.4 wt.%). Figure 16 shows that at 100% throttle, the 1 at.% alloy extruded at 2150°F (1175°C) gave the best recrystallization response albeit with a low grain aspect ratio. However, extrusions at 2050°F (1120°C) and 2150°F (1175°C) using only 35% press throttle gave a much better recrystallization response upon gradient annealing. Specifically, bar V71D gave a good elongated grain structure.

On this basis, alloy 20 containing 2 at.%Hf was extruded under the same conditions. However, the recrystallization response on gradient annealing was minimal as shown by bar V73A (Figure 16). Since poor elevated temperature mechanical properties were obtained on the 1 at.%Hf alloy (see Section 3.3), no further extrusions of the 2 at.% alloy were performed.

3.1.3.7 Effect of Titanium. Titanium at levels of 3 and 6 at.% (2.8 and 5.3 wt.%) was evaluated previously(5). A poor recrystallization response was obtained over a range of extrusion conditions. A further attempt was made to evaluate titanium, this time at a level of 1 at.% (0.9 wt.%). Figure 17 shows that a reasonable recrystallization response was obtained upon gradient annealing bars extruded with 100% press throttle at 2100°F (1150°C) and 2050°F (1120°C). Consequently, an additional extrusion at 2100°F (1150°C) and 35% press throttle was performed. As shown in Figure 17, a good elongated, recrystallized grain structure was obtained on gradient annealing.

3.1.3.8 Effect of Chromium. As described in the previous contract, the base alloy 2 composition: Ni-10 at.%Cr-17.5 at.%Al (Ni-9.8 wt.%Cr-8.9 wt.%Al-1.1 wt.% Y<sub>2</sub>O<sub>3</sub>) has rather poor sulfidation resistance. It is widely known that improved sulfidation resistance can be affected by increasing the chromium content of alloys. An increase in chromium content therefore was evaluated to determine its effect on recrystallization response evaluated. Figure 18 shows that excellent elongated grains were obtained for 15 at.%Cr bar extruded 18:1 at 2100°F (1150°C) and 35% press throttle. Higher extrusion speeds (100% press throttle) gave poorer structures.

Figure 19 shows that no significant recrystallization response was obtained for the 20 at.%Cr alloy extruded 18:1 at 2000°F (1095°C), 2050°F (1120°C), 2100°F (1150°C) or 2150°F (1175°C) using 100% press throttle. Extrusion at 2100°F using 35% press throttle did not produce any response either in this alloy.

3.1.4 Zone Annealing. Zone annealing was performed on alloys which showed promising recrystallization response upon gradient annealing. Specifically, zone annealing was completed successfully for alloys 2 (Ni-10 at.%Cr-17.5 at.%Al), 8 (2 at.%W), 9 (1 at.%Ta), 10 (3 at.%Ta), 12 (1 at.%Nb), 15 (1 at.%Mo), 16 (2 at.%Mo), 17 (5 at.%Co), 18 (10 at.%Co), 19 (1 at.%Hf), 22 (1 at.%Ti) and 23 (Ni-15 at.%Cr-17.5 at.%Al base). Optical micrographs showing typical zone annealed structures are given in Figures 20 through 29.

### 3.1.5 Mechanical Property Evaluation.

3.1.5.1 Stress Rupture. Preliminary stress rupture data at 2000°F (1095°C) was generated for alloys 2, 8, 9, 12, 15, 16, 19 and 23 using step loading tests (see Table IV). Alloys 15 (1 at.%Mo) and 16 (2 at.%Mo) gave the best properties; these alloys being able to sustain a maximum stress of 20 ksi (138 MPa) for 5 and 4 hours respectively. Alloys 8 (2 at.%W) and 12 (1 at.%Nb) can sustain a maximum stress of 18 ksi (124 MPa) for lives of 15 and 3-10 hours respectively.

Based on the step loading results, conventional stress rupture tests were run at 1400°F (760°C), 2000°F (1095°C), and 2100°F (1150°C) for alloys 9, 12, 15, 16 and 23. The results are given in Table V and plotted in Figures 30, 31 and 32. Using these data, the 100-hour and estimated 1000-hour rupture strengths have been determined and are shown in Table VI. Data on alloys 2 and 8 determined previously(5) are included for comparison. These data reveal the superior rupture strength of Mo-containing alloys at 1400°F (760°C), e.g., alloy 16 indicates a 100-hour rupture stress of 80 ksi (586 MPa). While at 2000°F, the W-containing alloy 8 has a 100-hour rupture stress of 19 ksi (131 MPa), compared with 17 ksi (117 MPa) for alloy 16. This pattern is repeated in the 2100°F (1150°C) stress rupture results, where the W-containing alloy 8 has a 100-hour rupture strength of 15 ksi (103 MPa), compared to 13 ksi (90 MPa) for the Mo-containing alloy 16. These results indicate that, of the elements examined, W, Mo, and to a lesser extent Nb, are the most effective elements for improving the rupture strength of the simple Ni-Cr-Al alloy.

Despite a good recrystallized grain structure, alloy 19 (1 at.%Hf) gave poor step load results indicative of structural instability and was, therefore, not tested further. This was confirmed by electron microscopy examination and, hence lower levels of Hf were used in the Series II alloys (see Section 3.2).

The density corrected 1000-hour and 300-hour rupture strengths of alloys 2, 8, 16 and 23 were determined (Tables VII and VIII, respectively), and plotted in Figures 33 and 34, respectively, for comparison with other materials systems(10).

~~3.1.5.1~~ 3.1.5.2 Tensile Test. The room temperature and 1400°F (760°C) tensile properties were determined for alloys 8, 9, 12, 15, 16, 19 and 23 (Table IX). The results indicate that the quaternary zone annealed alloys with either none or an additional, simple secondary heat treatment, have tensile properties at least comparable to the simple ternary alloy 2 and the widely-used nickel-base superalloys, e.g., IN-100, Mar M-200, and IN-792.

The poor performance of alloy 19 (1 at.%Hf) in stress rupture testing was confirmed by low elevated temperature tensile properties.

3.1.6 Gamma Prime Volume Fraction. A heat treatment study was conducted to determine the volume %  $\gamma'$  present in alloys 8, 9, 12, 15, 16, 17, 18, 19 and 23 at the intended operation temperature of 2000°F (1095°C). Specimens were, therefore, soaked and water quenched from 2000°F (1095°C). The samples were examined using replica electron microscopy and the %  $\gamma'$  volume fractions determined quantitatively from electron micrographs at 4900 and 7800X. The results are given in Table X and typical micrographs showing the  $\gamma'$  size, shape and distribution in Figures 35 through 40. It is apparent that the alloys retain a significant volume fraction of  $\gamma'$  at 2000°F (1095°C) approximating at least 50%.

3.1.7 Hot Corrosion Evaluation. Sulfidation corrosion tests were conducted in a low velocity burner rig of the G.E.-Lynn type using conventional superalloys as standards. The tests were conducted at 1700°F (930°C) over 168 hours with hourly cycles to room temperature. The flame composition was produced by burning JP-5 fuel (0.3% sulfur) at an air-to-fuel ratio of 30:1. The air was injected with 5 ppm seawater (ASTM Spec. D1141-52).

Table XI gives sulfidation test results on alloys 8, 9, 12, 15, 16, 17, 18, 19, 22 and 23. Also included is previously determined data(5) on alloy 2 (Ni-9.9 wt.%Cr-9.0 wt.%Al-1.1 wt.%Y<sub>2</sub>O<sub>3</sub>). It is readily apparent that raising the chromium level in the base ternary composition to 15 wt.% reduced the sulfidation attack by approximately 50% for equivalent exposure times.



The corrosion resistance of all the experimental alloys was superior to IN-713C and IN-100, although alloy 19 (3.3 wt.%Hf) was suspect (see Discussion Section 3.3). Raising the chromium level in the Ni-Cr-Al base alloy significantly improved the corrosion resistance in relation to the highly corrosion resistant IN-738. Further improvements were expected to accrue from other elemental additions such as W and Ti in the Series II alloys.

High temperature cyclic oxidation test data were determined on duplicate samples of several alloys, as shown in Table XII. These tests were performed at 2012°F (1100°C) for 504 hours with daily cycles to room temperature. The atmosphere was air-5% water vapor at low velocity (15 in.<sup>3</sup> min.<sup>-1</sup>/250 cc. min.<sup>-1</sup>). The results show that all the alloys have excellent oxidation resistance. The descaled weight losses were generally less than observed for IN-100 and far less than that observed for alloys 713C, 713LC, and IN-738. The extremely high oxidation resistance exhibited by the experimental alloy systems make them ideally suited for high temperature turbine applications.

### 3.2 Evaluation of Series II Alloys

#### 3.2.1 Powder Processing of Series II Alloys.

Table XIII gives the compositions derived from a statistically designed set of alloys which embodies those elements found not to impede recrystallization to an elongated grain structure of high grain aspect ratio, while maintaining a high volume fraction of  $\gamma'$  precipitate. The alloys were prepared by mechanical alloying in the same manner as the Series I alloys. Each powder batch was characterized by chemical and screen analysis and by metallographic examination. The results of these powder characterization studies are given in Table XIV. It should be noted that the powder size of the more complex Series II alloys was generally finer than the Series I alloys. Microstructural examination indicated that the finer powder was still as well processed as the relatively coarser powders obtained from Series I alloys.

3.2.2 Thermomechanical Processing. Mechanically alloyed powder of each alloy composition was prepared and canned for extrusion as described for Series I alloys. A minimum of four cans was prepared for each composition listed in Table XIII. The extrusion conditions employed for consolidation of powder are detailed in Table XV. Following extrusion, the alloys were tested for recrystallization response by gradient annealing.

3.2.3 Gradient Annealing Studies. Alloy 25 did not show any significant recrystallization response even at temperatures close to the melting point (Figure 41). Consequently, the range of extrusion conditions was widened



to encompass a temperature range of 1850°F (1010°C)-2150°F (1175°C), extrusion ratios of 18 and 30:1, and extrusion press throttle settings (i.e., extrusion ram speed) of 35% and 100%. However, even these variations failed to produce any significant recrystallization response in alloys 25-33. Figure 42 shows gradient anneal bars of Alloy 27 which typify the negative response of these alloys.

In order to verify that processing conditions had not changed in any significant way from those used for Series I alloys, a batch of Alloy 2 powder was prepared and processed. The characterization of these heats was very similar to that of previous heats of this alloy, indicating that the poor recrystallization response of Series II alloys tested to date was attributable to composition rather than processing effects. It was apparent that, whereas the individual quaternary additions of W, Mo, Co, Hf, Ta, Nb and Ti were amenable to directional recrystallization, the response conditions in more complex alloys containing combinations of these elements was more critical.

Consequently, leaner compositions based on the promising W-containing quaternary Alloy 8 were investigated in Alloys 34 to 36. However, recrystallization response was again poor indicating a critical compositional effect such as transposition into a phase field other than  $\gamma + \gamma'$ , e.g., introduction of  $\beta$  phase.

### 3.3 General Discussion

Work on the effect of quaternary alloying elements on the recrystallization behavior of the basic Ni-9.9 wt.%Cr-9.0 wt.%Al-1.1 wt.%Y<sub>2</sub>O<sub>3</sub> has been completed. Observations on recrystallization response indicated that high levels of the group IVB and VB  $\gamma'$  forming elements, Ti, Nb, Ta, and Hf, impede the development of coarse elongated grains in the base alloy composition. Solid solution elements from group VIB and group VIII, i.e., Cr, Mo, W and Co do not impede the development of elongated grain structures. It was also determined that slower ram speeds could enhance the recrystallization response.

Despite the excellent recrystallization response of alloy 19 (1 at.%Hf) resulting from slower extrusion speeds, the elevated temperature mechanical and sulfidation resistance were poor. The cause of this structural weakness was not identified positively. It may be due to the presence of a deleterious Hf-rich phase resulting from excess Hf. Consequently, the Hf content was reduced to 0.5 at.% (approximately 1.6 wt.%) in the relevant Series II alloys.

The development of an elongated grain structure at higher chromium levels was particularly important, since the Ni-15 Wt.%Cr-9 wt.%Al-1.1 wt.%Y<sub>2</sub>O<sub>3</sub> material (Alloy 23) had

effectively double the corrosion resistance of the original alloy with 10 wt.%Cr (Alloy 2). Alloy 23 was therefore adopted as the base composition for the Series II alloys.

Specific rupture strength properties of the respective W and Mo-containing alloys (Figures 33 and 34) showed excellent promise for further development. Within the typical operating stress regime of a turbine blade, these high volume fraction  $\gamma'$  ODS alloys currently display up to 250°F (120°C) and 150°F (66°C) temperature advantages over the DS Mar M-200 + Hf and DS  $\gamma/\gamma'-\alpha$  eutectic alloys, respectively.

Moreover, even the simple quaternary high volume %  $\gamma'$  Ni-Cr-Al-W Alloy 8 (density 8.15 gm/cm<sup>3</sup>) compares very favorably with the complex and considerably more developed alloy MA6000E (density 8.11 gm/cm<sup>3</sup>) on rupture strength. The latter has (11) optimum 300- and 1000-hour specific rupture strengths at 2000°F (1095°C) of 22 ksi (151 MPa) and 21 ksi (145 MPa), respectively, while Alloy 8 has values of 19 ksi (131 MPa) and 18 ksi (124 MPa). This is a very significant indication of the excellent potential that exists for developing the properties of these high volume %  $\gamma'$  Ni-Cr-Al base alloys beyond the levels achieved in alloy MA6000E.

Raising the chromium level in the simple Ni-Cr-Al ternary composition to 15 wt.% (Alloy 23) was justified by the need to improve the hot corrosion resistance of the base Alloy 2. While the tensile strength was also improved, the initial slight reduction in rupture strength would be minimized by subsequent development of an optimized structure. Also, electron microscopy reaffirmed that the high volume fraction of  $\gamma'$  precipitate had been retained in the quaternary alloys and, in particular, the new high chromium base Alloy 23 (Figure 40). Consequently, quaternary and higher order additions of elements, found beneficial to Alloy 2, made on an atomic substitutional basis were expected to yield similar results in Alloy 23 (i.e., Series II compositions). However, as the results indicate, this was not the case. It was established in the previous contract(5) that the structure of Alloy 2 at 2100°F (1150°C) was not in the  $\gamma$  phase field, as reported by Taylor(12), but in the  $\gamma + \gamma'$  phase field. Increasing the chromium content of Alloy 2 moves the composition locus point towards the  $\beta$ -containing phase field. This trend can lead to problems, since it was found(5) that  $\beta$ -containing alloys apparently did not respond to the normal mechanical alloying synthesis. Very fine powders typifying an extreme grinding regime were produced. The composition of Alloy 23 lies close to the  $\beta$ -containing phase field solvus lines. Raising the chromium content of the

Ni-Cr-Al ternary to 20 wt.%, as in Alloy 24, places the composition locus point just inside a  $\beta$ -containing phase field: This alloy did not respond to gradient annealing (Figure 19) and x-ray diffraction studies confirmed the presence of  $\beta$  phase. Therefore, it is possible to infer that quaternary and higher order element additions to Alloy 23 could displace the composition locus point into the  $\gamma + \gamma' + \beta$  or  $\gamma + \beta$  phase fields, particularly since there is some doubt now as to where the solvus lines actually lie in this region of the Ni-Cr-Al phase diagram. The fact that Series II mechanically alloyed powders were finer than Series I, tends to support this hypothesis as  $\beta$ -containing alloys were found to produce very fine powders(5). Subsequent x-ray diffraction studies on selected Series II alloys confirmed the presence of  $\beta$  phase.

It should also be noted that the additions, although added on an atomic substitutional basis, do not necessarily partition entirely to either the  $\gamma$  or  $\gamma'$  phase but to both depending on their respective partition coefficients. Recent work(11) indicates, for example, that in MA6000E, there is more tungsten in the  $\gamma'$  and tantalum in the  $\gamma$  phase than would normally be expected.

Alloys 34 to 35 were made leaner in composition to investigate the processing response of materials lying closer in the phase diagram to the Alloy 23. These compositions showed promise in as much as the mechanically alloyed powders had fewer fines, approaching the more desirable lower levels achieved in Alloy 23 (Ref. Tables II and XIV). However, these alloys failed to recrystallize on gradient annealing.

These present Series II results notwithstanding, it is felt that additional areas of research are open for exploration to develop a high strength high volume fraction  $\gamma'$  alloy. One area is further adjustments in alloy compositions. Specifically, the aluminum content of the alloys could be lowered to a level just sufficient to eliminate the formation of any undesirable  $\beta$ -phase but still retaining a high volume %  $\gamma'$ . The chromium level would be fixed at 15 wt.% to retain the good hot corrosion resistance.

Other important areas for property improvement are processing, thermomechanical working and heat treatment. In particular, experiments were begun on investigating the effect of extrusion preheat time, as there were promising indications that shorter times were beneficial to the recrystallization response of finer mechanically alloyed powder. In addition, the use of secondary thermomechanical working operations (e.g., hot rolling of extruded bar) should improve the recrystallization response and properties of the selected alloys. These aspects can be addressed once an alloy base has been identified.



#### IV. SUMMARY

This work has identified the nature and level of quaternary addition elements that are beneficial to the directional recrystallization response and properties of oxide dispersion strengthened, high volume fraction  $\gamma'$  Ni-Cr-Al alloys made by mechanical alloying. In particular, tungsten and molybdenum additions to the Ni-10 wt.%Cr-9 wt.%Al-1.1 wt.%Y<sub>2</sub>O<sub>3</sub> base alloy respectively evolved simple quaternary alloys with significant high temperature strength advantages over DS Mar M-200 + Hf and a current DS  $\gamma/\gamma'-\alpha$  eutectic. Furthermore, there is a significant indication that the properties of these experimental alloys may be developed beyond the levels achieved in the complex and considerably more developed alloy MA6000E.

Electron microscopy has reaffirmed the volume fraction of  $\gamma'$  present at the intended use temperature ( $\approx$  2000°F/1095°C). The sulfidation resistance of the experimental alloys was superior to IN-713C and IN-100. Raising the chromium level in the simple Ni-Cr-Al base alloy to 15 wt.% reduced the sulfidation attack by 50%. Oxidation resistance of the experimental alloys was excellent.

The data from the simple ternary and quaternary alloys (Series I) was used to design more complex alloys based on atomic substitution in the more corrosion resistant Ni-15 wt.%Cr-9 wt.%Al-1.1 wt.%Y<sub>2</sub>O<sub>3</sub> alloy. The alloys contained combinations of W, Co, Mo, Ta, Nb, Ti, and Hf. However, these alloys did not yield the required directionally recrystallized grain structure on gradient annealing. The poor structural response is attributed to the presence of  $\beta$  phase. Additional minor compositional modifications to overcome this problem are outlined together with other important areas for property improvement.

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TABLE I

SERIES I ALLOYS (COMPOSITIONS IN ATOMIC AND WEIGHT %)

Alloy No.		Atomic %*				Weight %				
		Ni	Cr	Al	X	Ni	Cr	Al	X	Y <sub>2</sub> O <sub>3</sub>
V60	2	72.5	10	17.5	--	80.2	9.8	8.9	--	1.1
V37	8	70.5	10	17.5	2 W	74.4	9.4	8.5	6.6 W	1.1
V42	9	72.5	10	16.5	1 Ta	78.0	9.5	8.1	3.3 Ta	1.1
V44	10	72.5	10	14.4	3 Ta	73.7	9.0	6.8	9.3 Ta	1.1
V46	11	72.5	10	11.5	6 Ta	69.4	8.3	4.9	17.4 Ta	1.1
V49	12	72.5	10	16.5	1 Nb	79.2	9.7	8.3	1.7 Nb	1.1
V51	13	72.5	10	14.5	3 Nb	77.3	9.4	7.1	5.1 Nb	1.1
V53	14	72.5	10	11.5	6 Nb	74.6	9.1	5.4	9.8 Nb	1.1
V59	15	71.5	10	17.5	1 Mo	78.6	9.7	8.8	1.8 Mo	1.1
V63	16	70.5	10	17.5	2 Mo	76.9	9.7	8.8	3.6 Mo	1.1
V66	17	67.5	10	17.5	5 Co	74.7	9.8	8.9	5.5 Co	1.1
V68	18	62.5	10	17.5	10 Co	69.1	9.8	8.9	11.1 Co	1.1
V71	19	71.5	10	17.5	1 Hf	77.3	9.6	8.7	3.3 Hf	1.1
V73	20	70.5	10	17.5	2 Hf	74.6	9.4	8.5	6.4 Hf	1.1
V75	21	68.5	10	17.5	4 W	69.1	8.9	8.1	12.7 W	1.1
V78	22	72.5	10	16.5	1 Ti	79.9	9.8	8.4	.9 Ti	1.1
V81	23	67.5	15	17.5	--	75.2	14.8	8.9	--	1.1
V83	24	62.5	20	17.5	--	70.0	19.9	9.0	--	1.1

\*Excluding added Y<sub>2</sub>O<sub>3</sub>



TABLE II

## POWDER CHARACTERIZATION OF SERIES I ALLOYS

Alloy No.	Batch No.	Chemistry, Wt. %			Screen Analysis, Mesh Size, %							
		O	N	C	+40	-40/+80	-80/+100	-100/+140	-140/+200	-200/+325	-325	
8	V37*	.56	.063	.057	15.0	44.6	9.5	11.4	10.2	6.1	3.2	
8	V38	.52	.054	.051	13.9	52.5	11.3	11.3	7.3	2.6	1.1	
9	V42	.42	.032	.060	17.9	42.4	10.8	10.7	9.0	5.5	3.7	
9	V43	.54	.053	.066	26.6	46.1	8.0	8.0	5.3	3.3	2.7	
10	V44	.56	.049	.062	18.5	27.3	9.6	14.6	15.5	9.5	5.0	
10	V45	.61	.053	.063	21.1	29.7	8.8	12.9	14.3	8.6	4.6	
11	V46	.59	.054	.057	12.9	24.0	5.3	10.9	16.5	17.6	12.8	
11	V47	.57	.044	.058	9.3	22.9	5.4	11.0	17.4	19.4	14.6	
12	V49	.58	.062	.066	25.6	30.0	8.9	11.5	11.4	6.6	6.0	
12	V50	.53	.056	.068	24.5	45.2	8.4	8.2	6.0	4.0	3.7	
13	V51	.51	.073	.069	11.7	24.1	6.1	10.3	16.7	18.1	13.0	
13	V52	.57	.064	.066	18.8	36.0	9.5	11.9	10.1	7.9	5.8	
14	V53	.58	.096	.063	9.6	27.0	5.5	10.3	15.5	16.9	15.2	
14	V54	.60	.086	.062	9.2	27.9	5.4	8.8	13.9	16.9	17.9	
15	V59	.65	.051	.065	28.8	37.8	8.7	11.7	7.7	3.4	1.9	
15	V62	--	--	--	32.4	38.5	8.5	9.0	6.3	3.3	2.0	
2	V60	.40	.053	.070	18.4	31.9	9.8	13.5	12.7	8.0	5.7	
2	V61	.55	.056	.068	19.5	40.7	9.3	10.2	9.3	6.2	4.8	
16	V63	.53	.053	.061	35.6	40.2	7.4	7.2	5.6	2.6	1.4	
16	V64	.48	.067	.059	17.2	30.3	12.4	17.4	15	5.7	2.0	
17	V66	.47	.056	.067	15.0	25.6	8.9	13.3	16.3	12.1	8.8	
17	V67	.58	.055	.067	12.4	39.6	11.4	11.1	10.1	7.7	7.7	

TABLE II (CONTINUED)

Alloy No.	Batch No.	Chemistry, Wt. %			Screen Analysis, Mesh Size, %							
		O	N	C	+40	-40/+80	-80/+100	-100/+140	-140/+200	-200/+325	-325	
18	V68	.15	.046	.066	12.5	37.9	11.3	12.2	11.5	7.8	6.8	
18	V69	.25	.041	.07	13.0	46.3	10.3	10.2	8.8	6.0	5.3	
19	V71	.40	.068	.071	29.6	25.9	6.5	11.1	13.4	8.8	4.7	
19	V72	.66	.070	.067	31.2	26.4	5.7	8.6	11.3	10.2	6.6	
20	V73	.63	.070	.068	13.9	27.2	8.7	12.9	13.3	13.4	10.6	
21	V75	.33	.057	.060	9.4	41.3	11.4	12.2	10.2	9.1	7.4	
21	V76	.35	.061	.061	10.6	30.4	10.1	12.6	14.5	11.9	9.9	
22	V78	.40	.053	.069	13.3	33.3	10.1	12.2	12.5	10.1	8.5	
22	V79	.30	.057	.070	19.9	36.9	10.9	10.6	9.9	6.9	4.9	
23	V81	.29	.066	.064	10.1	36.1	12.0	13.5	12.4	8.2	7.7	
23	V82	.30	.068	.064	13.4	42.7	9.3	10.5	10.1	7.5	6.5	
24	V83	.27	.075	.064	8.5	39.1	10.7	13.2	11.2	8.6	8.7	
24	V84	.21	.084	.064	12.1	31.3	9.4	12.0	11.8	11.9	11.5	

\*The batch number V37 was assigned to the total of the combined alloy 8 batches V37 and V38 which were cone blended. Likewise, V42, V44, V46, etc. were assigned to the two cone blended batches of alloys 9, 10, 11, etc., respectively.

TABLE III  
EXTRUSION CONDITIONS OF  
SERIES I ALLOYS

<u>Alloy No.</u>	<u>Billet No.</u>	<u>Temperature °F (°C)</u>	<u>Ratio</u>	<u>Ram Speed* in/sec (cm/sec)</u>
2	V21-A	1750 (955)	26	5.9 (15.0)
	V21-B	1950 (1065)	30	4.9 (12.4)
	V21-C	2150 (1175)	36	3.7 (9.4)
	V21-D	2150 (1175)	28	7.6 (19.3)
	V21-E	2150 (1175)	18	6.9 (17.5)
	V21-F	1900 (1040)	18	5.0 (12.7)
	V21-G	2050 (1120)	30	8.2 (20.8)
	V21-H	2150 (1175)	50	7.5 (19.1)
	V21-I	2200 (1205)	50	5.4 (13.7)
8	V37-A	2150 (1175)	55	2.0 (5.1)**
	V37-B	2150 (1175)	20	4.0 (10.2)**
	V37-C	2050 (1120)	55	3.0 (7.6)
	V37-D	2050 (1120)	20	12.1 (30.7)
	V37-E	2150 (1175)	30	11.7 (29.7)
	V37-F	2050 (1120)	20	3.2 (8.1)**
9	V42-A	2100 (1150)	19	17.8 (45.2)
	V42-B	2050 (1120)	19	12.9 (32.8)
	V42-C	2000 (1095)	18	11.3 (28.7)
	V42-D	1950 (1065)	18	NO RECORD
	V42-E	2150 (1175)	18	13.3 (33.8)
	V42-F	2200 (1205)	18	14.1 (35.8)
	V42-G	2100 (1150)	18	NO RECORD**
10	V44-A	2100 (1150)	18	NO RECORD
	V44-B	2050 (1120)	18	13.3 (33.8)
	V44-C	2000 (1095)	18	12.5 (31.8)
	V44-D	1950 (1065)	18	11.7 (29.7)
	V44-E	2150 (1175)	18	12.9 (32.8)
	V44-F	2200 (1205)	18	14.1 (35.8)
11	V46-A	2200 (1205)	18	13.7 (34.8)
	V46-B	2150 (1175)	18	13.3 (33.8)
	V46-C	2100 (1150)	18	12.2 (31.0)
	V46-D	2050 (1120)	18	9.9 (25.1)
	V46-E	2050 (1120)	36	NO RECORD
	V46-F	1950 (1065)	18	11.5 (29.1)

\*100% press throttle

\*\* 35% press throttle



TABLE III (CONTINUED)

<u>Alloy No.</u>	<u>Billet No.</u>	<u>Temperature</u> <u>°F (°C)</u>	<u>Ratio</u>	<u>Ram Speed*</u> <u>in/sec (cm/sec)</u>
12	V49-A	2200 (1205)	18	13.7 (34.8)
	V49-B	2150 (1175)	18	14.1 (35.8)
	V49-C	2100 (1150)	18	12.9 (32.8)
	V49-D	2050 (1120)	18	11.3 (28.7)
	V49-E	2050 (1120)	36	NO RECORD
	V49-F	2000 (1095)	18	12.5 (31.6)
13	V51-A	2200 (1205)	18	NO RECORD
	V51-B	2150 (1175)	18	NO RECORD
	V51-C	2100 (1150)	18	11.3 (28.7)
	V51-D	2050 (1120)	18	4.0 (10.2)
	V51-E	2050 (1120)	36	NO RECORD
	V51-F	1950 (1065)	18	12.5 (31.6)
14	V53-A	2200 (1205)	18	NO RECORD
	V53-B	2150 (1175)	18	NO RECORD
	V53-C	2100 (1150)	18	11.3 (28.7)
	V53-D	2050 (1120)	18	4.8 (12.2)
	V53-E	2050 (1120)	36	NO RECORD
	V53-F	1950 (1065)	18	12.1 (30.6)
15	V59-A	2100 (1150)	18	14.9 (37.7)
	V59-B	2050 (1120)	18	12.1 (30.6)
	V59-C	2000 (1095)	18	11.7 (29.6)
	V59-D	2050 (1120)	18	NO RECORD
	V59-E	2050 (1120)	36	8.5 (21.5)
	V59-F	2050 (1120)	16	13.9 (35.2)
2	V60-A	2200 (1205)	50	10.1 (25.6)
	V60-B	2150 (1175)	50	8.5 (21.5)
	V60-C	2050 (1120)	36	NO RECORD
	V60-D	2050 (1120)	18	13.7 (34.7)
	V60-E	2100 (1150)	18	14.3 (36.2)
	V60-F	2075 (1135)	18	NO RECORD
16	V63-A	2100 (1150)	18	NO RECORD
	V63-B	2050 (1120)	18	NO RECORD
	V63-C	2000 (1095)	18	NO RECORD
17	V66-A	2100 (1150)	18	NO RECORD
	V66-B	2050 (1120)	18	NO RECORD
	V66-C	2000 (1095)	18	NO RECORD
	V66-D	2100 (1150)	18	4.1 (10.4)**

\*100% press throttle  
 \*\* 35% press throttle

TABLE III (CONTINUED)

<u>Alloy No.</u>	<u>Billet No.</u>	<u>Temperature °F (°C)</u>	<u>Ratio</u>	<u>Ram Speed* in/sec (cm/sec)</u>
18	V68-A	2100 (1150)	18	NO RECORD
	V68-B	2050 (1120)	18	NO RECORD
	V68-C	2000 (1095)	18	NO RECORD
	V68-D	2100 (1150)	18	4.2 (10.7)**
19	V71-A	2050 (1120)	18	10.1 (25.6)
	V71-B	2100 (1150)	18	NO RECORD
	V71-C	2150 (1175)	18	15.1 (38.3)
	V71-D	2150 (1175)	18	NO RECORD**
	V71-E	2050 (1120)	18	NO RECORD**
20	V73-A	2150 (1175)	18	4.3 (10.9)**
21	V75-A	2050 (1120)	18	NO RECORD
	V75-B	2100 (1150)	18	12.1 (30.6)
	V75-C	2150 (1175)	18	14.1 (35.7)
	V75-D	2150 (1175)	18	4.0 (10.2)**
	V75-F	2050 (1120)	18	NO RECORD**
22	V78-A	2150 (1175)	18	NO RECORD
	V78-B	2100 (1150)	18	15.1 (38.2)
	V78-C	2050 (1120)	18	13.5 (34.2)
	V78-D	2100 (1150)	18	4.2 (10.7)**
23	V81-A	2150 (1175)	18	16.5 (41.7)
	V81-B	2100 (1150)	18	15.5 (39.2)
	V81-C	2050 (1120)	18	14.5 (36.7)
	V81-D	2100 (1150)	18	NO RECORD**
24	V83-A	2150 (1175)	18	16.5 (41.7)
	V83-B	2100 (1150)	18	15.1 (38.2)
	V83-C	2050 (1120)	18	14.7 (37.2)
	V83-D	2000 (1095)	18	NO RECORD
	V83-E	2100 (1150)	18	4.0 (10.2)&&

\*100% press throttle  
 \*\*30-40% press throttle

TABLE IV

## PRELIMINARY STRESS RUPTURE RESULTS (STEP LOAD)

Alloy No.	Bar No.	Zone Anneal		Temperature °F (°C)	Speed ips (cm/s)	Stress		Life (hr)	Elong. (%)	R.A. (%)
		Temperature °F (°C)	Speed ips (cm/s)			ksi	(MPa)			
2	V60D-1	2290 (1255)	2.8 ( 7.1)	2000 (1095)		14	( 96.5)	24	STEP LOAD	
						16	(110.0)	24	STEP LOAD	
						18	(124.0)	5.4	4.3	2.5
8	V37B-1	2245 (1230)	3.0 ( 7.6)	2000 (1095)		14	( 96.5)	24	STEP LOAD	
						16	(110.0)	24	STEP LOAD	
						18	(124.0)	15.6	4.4	2.5
9	V42A-1	2390 (1310)	5.3 (13.5)	2000 (1095)		14	( 96.5)	23	STEP LOAD	
						16	(110.0)	9.8	1.3	2.9
9	V42A-2	2310 (1265)	5.3 (13.5)	2000 (1095)		14	( 96.5)	23	STEP LOAD	
						16	(110.0)	3.6	1.3	1.5
12	V49D-1	2365 (1295)	5.3 (13.5)	2000 (1095)		14	( 96.5)	24	STEP LOAD	
						16	(110.0)	24	STEP LOAD	
						18	(124.0)	10.3	1.3	1.5
12	V49D-2	2355 (1290)	5.3 (13.5)	2000 (1095)		14	( 96.5)	24	STEP LOAD	
						16	(110.0)	24	STEP LOAD	
						18	(124.0)	5.5	3.8	7.2
12	V49D-3	2355 (1290)	5.3 (13.5)	2000 (1095)		14	( 96.5)	24	STEP LOAD	
						16	(110.0)	24	STEP LOAD	
						18	(124.0)	3.4	1.3	2.8
15	V59B-1	2290 (1255)	2.8 ( 7.1)	2000 (1095)		14	( 96.5)	24	STEP LOAD	
						16	(110.0)	24.2	STEP LOAD	
						18	(124.0)	24	STEP LOAD	
						20	(138.0)	5.2	2.9	1.3



TABLE IV (CONTINUED)

Alloy No.	Bar No.	Zone Anneal Temperature °F (°C)	Zone Anneal Speed ips (cm/s)	Temperature °F (°C)	ksi	Stress (MPa)	Life (hr)	Elong. (%)	R.A. (%)
16	V63B-1	2315 (1270)*	3.0 ( 7.6)	2000 (1095)	14	( 96.5)	24	STEP LOAD	
					16	(110.0)	24	STEP LOAD	
					18	(124.0)	24	STEP LOAD	
					20	(138.0)	3.8	1.4	1.3
19	V71D-1	2340 (1280)*	3.0 ( 7.6)	2000 (1095)	14	( 96.5)	Nil	7.2	5.0
					10	( 69.0)	0.1	8.5	8.6
					8	( 55.0)	0.4	4.3	2.5
23	V81D-1	2360 (1295)*	3.0 ( 7.6)	2000 (1095)	14	( 96.5)	24.0	1.5	1.3

\*Post zone anneal heat treatment given: 1/2 hr/zone anneal temperature/AC.

TABLE V

1400°F (760°C), 2000°F (1095°C) AND 2100°F (1150°C) STRESS  
RUPTURE RESULTS FOR ALLOYS 2, 8, 9, 12, 15, 16, AND 23

Alloy No.	Bar No.	Zone Anneal		Temperature °F (°C)	Zone Anneal Speed ips (cm/s)	Temperature		Stress		Life (hr)	Elong. (%)	R.A. (%)
		Temperature °F (°C)	Speed ips (cm/s)			°F (°C)	°F (°C)	ksi	(MPa)			
9	V42A-1	2390 (1310)	5.3 (13.5)	1400 (760)	70	(482.5)	57.9	2.9	2.5			
	V42A-3	2410 (1320)	5.3 (13.5)	1400 (760)	85	(586.0)	4.1	2.9	2.5			
	V42A-3	2410 (1320)	5.3 (13.5)	2000 (1095)	14	(96.5)	140.8	1.5	1.3			
					16	(110.0)	22.9	1.4	1.3			
12	V42A-3	2410 (1310)	5.3 (13.5)	2100 (1150)	10	(69.0)	281.3	Nil	Nil			
	V42A-1	2390 (1320)	5.3 (13.5)	2100 (1150)	12	(83.0)	11.4	2.9	1.3			
	V49D-2	2355 (1290)	5.3 (13.5)	1400 (760)	70	(482.5)	168.7	2.9	2.5			
	V49D-3	2355 (1290)	5.3 (13.5)	1400 (760)	85	(586.0)	18.4	4.3	3.8			
15	V49D-2	2355 (1290)	5.3 (13.5)	2000 (1095)	14	(96.5)	395	Nil	Nil			
	V49D-3	2355 (1290)	5.3 (13.5)	2000 (1095)	16	(110.0)	52.5	Nil	1.3			
	V49D-2	2355 (1290)	5.3 (13.5)	2100 (1150)	10	(69.0)	108.5	Nil	1.3			
						(83.0)	92.2	1.5	1.3			
15	V59B-3	2275 (1245)	2.8 (7.1)	1400 (760)	75	(517.0)	97.3	3.0	1.3			
					85	(586.0)	21.2	4.3	2.5			
	V59B-3	2275 (1245)	2.8 (7.1)	2000 (1095)	16	(110.0)	223.4	Nil	1.3			
					20	(138.0)	10.2	8.7	2.5			

TABLE V (CONTINUED)

Alloy No.	Bar No.	Zone Anneal Temperature °F (°C)	Zone Anneal Speed ips (cm/s)	Temperature °F (°C)	Stress		Life (hr)	Elong. (%)	R.A. (%)
					ksi	(MPa)			
16	V63B-1	2315 (1270)*	3.0 (7.6)	1400 (760)	75	(517.0)	173.3	4.2	2.5
					85	(586.0)	54.0	2.9	1.3
	V63B-1	2315 (1270)*	3.0 (7.6)	2000 (1095)	16	(110.0)	177.6	Nil	1.3
23	V63B-1	2315 (1270)*	3.0 (7.6)	2100 (1150)	20	(138.0)	8.4	2.9	1.3
					14	(96.5)	17.8	4.3	2.5
	V63B-1	2315 (1270)*	3.0 (7.6)	2100 (1150)	16	(110.0)	0.9	4.3	2.5
23	V81D-1	2360 (1295)*	3.0 (7.6)	1400 (760)	70	(482.5)	79.1	3.0	1.8
					85	(586.0)	9.6	2.9	1.3
	V81D-1	2360 (1295)*	3.0 (7.6)	2000 (1095)	12	(83.0)	303.1	4.3	2.5
2	V81D-1	2360 (1295)*	3.0 (7.6)	2100 (1150)	14	(96.5)	24.0	1.5	1.3
					8	(55.0)	264.4	4.3	3.8
	V81D-1	2360 (1295)*	3.0 (7.6)	2100 (1150)	10	(69.0)	17.6	7.2	6.2
2	V21I	2385 (1310)	5.3 (13.5)	1400 (760)	85	(586.0)	20.1	2.5	4.3
					70	(482.5)	156.1	3.8	7.2
	V21G	2375 (1300)	5.3 (13.5)	2000 (1095)	18	(124.0)	1.6	1.3	2.9
2	V21G	2375 (1300)*	5.3 (13.5)	2000 (1095)	16	(110.0)	61.0	2.5	5.8
					15	(103.0)	44.7	2.5	8.5
	V21G	2375 (1300)	5.3 (13.5)	2100 (1150)	14	(96.5)	769.4	2.5	4.3
2	V21G	2375 (1300)	5.3 (13.5)	2100 (1150)	12	(83.0)	53.4	5.0	8.5
					10	(69.0)	1005.7	3.8	8.7

TABLE V (CONTINUED)

Alloy No.	Bar No.	Zone Anneal Temperature °F (°C)	Zone Anneal Speed ips (cmps)	Temperature °F (°C)	Stress ksi (MPa)	Life (hr)	Elong. (%)	R.A. (%)
8	V37F	2275 (1250)	5.3 (13.5)	1400 (760)	80 (552.0)	7.5	2.5	2.9
					75 (517.0)	90.6	1.3	1.4
	V37F	2275 (1250)	5.3 (13.5)	2000 (1095)	20 (138.0)	20.2	2.5	3.0
					18 (124.0)	612.5	1.3	1.5
	V37F	2275 (1250)	5.3 (13.5)	2100 (1150)	16 (110.0)	25.2	2.5	2.9
					14 (96.5)	222.3	1.3	2.9

\*Post zone anneal heat treatment given: 1/2 hr/zone anneal temperature/AC.



TABLE VI

RUPTURE STRENGTHS OF ALLOYS 2,8,9,12,15,16 AND 23

Alloy No.	Bar No.	Temperature °F (°C)	Strength, ksi (MPa)			
			100 Hour		1000 Hour*	
2	V21I	1400 (760)	73	(503)	59	(407)
	V21G	2000 (1095)	15	(103)	14	(96.5)
	V21G	2100 (1150)	11.5	(79)	10	(69)
8	V37F	1400 (760)	73	(503)	71	(490)
	V37F	2000 (1095)	19	(131)	18	(124)
	V37F	2100 (1150)	15	(103)	13	(90)
9	V42A	1400 (760)	67	(762)	58	(400)
	V42A	2000 (1095)	14	(96.5)	12	(83)
	V42A	2100 (1150)	10.5	(72)	9	(62)
12	V49D	1400 (760)	73	(503)	60	(414)
	V49D	2000 (1095)	15	(103)	13.5	(93)
15	V59B	1400 (760)	75	(517)	63	(434)
	V59B	2000 (1095)	17	(117)	14.5	(100)
16	V63B	1400 (760)	80	(586)	64	(441)
	V63B	2000 (1095)	17	(117)	14	(96.5)
	V63B	2100 (1150)	13	(90)	12	(83)
23	V81D	1400 (760)	70	(483)	57	(393)
	V81D	2000 (1095)	13	(90)	11	(76)
	V81D	2100 (1150)	9	(62)	7	(48)

\*Strength levels at 1000 hour test duration are estimated values.

TABLE VII

1000 HOUR SPECIFIC RUPTURE STRENGTH-  
TEMPERATURE DATA FOR FIGURE 1

Alloy	Density $\rho$ , lb/in <sup>3</sup> (g/cm <sup>3</sup> )	Temperature °F (°C)	Stress, $\sigma$ ksi (MPa)	$\sigma/\rho$ in x 10 <sup>3</sup> (cm x 10 <sup>3</sup> )
*DS Mar M-200 + Hf	0.312 (8.63)	1400 (760)	92 (634)	295 (749)
		1600 (870)	48 (749)	154 (391)
		1800 (980)	16.2 (112)	52 (132)
*DS eutectic $\gamma/\gamma'$ - $\alpha$	0.307 (8.50)	1400 (760)	102 (703)	322 (843)
		1600 (870)	57 (393)	186 (472)
		1800 (980)	25.5 (176)	83 (211)
		2000 (1095)	11.5 (79)	37 (94)
		2100 (1150)	6.5 (45)	21.2 (54)
Alloy 2	0.275 (7.60)	1400 (760)	59 (407)	214 (543)
		1800 (980)	22.5 (155)	82 (208)
		1900 (1040)	17.3 (119)	63 (160)
		2000 (1095)	13.1 (90)	47.5 (121)
		2100 (1150)	9.6 (66)	35 (89)
Alloy 8	0.295 (8.15)	1400 (760)	71 (490)	241 (612)
		2000 (1095)	17.9 (123)	61 (155)
		2100 (1150)	12.9 (89)	44 (112)
Alloy 16	0.274 (7.57)	1400 (760)	65 (448)	237 (602)
		2000 (1095)	14 (96.5)	51.1 (130)
		2100 (1150)	12 (83)	43.8 (111)

\*From Reference 10.

TABLE VIII  
300 HOUR SPECIFIC RUPTURE STRENGTH-  
TEMPERATURE DATA FOR FIGURE 7

Alloy	Density $\rho$ , lb/in <sup>3</sup> (g/cm <sup>3</sup> )	Temperature °F (°C)	Stress, $\sigma$ ksi (MPa)	$\sigma/\rho$ in x 10 <sup>3</sup> (cm x 10 <sup>3</sup> )
*DS Mar M-200 + Hf	0.312 (8.63)	1400 (760)	100 (689.5)	320.5 (814)
		1600 (870)	57 (393)	182.7 (464)
		1800 (980)	22 (152)	70.5 (179)
*DS eutectic $\gamma/\gamma'$ - $\alpha$	0.307 (8.50)	1400 (760)	101 (696)	328.9 (835)
		1600 (870)	67 (462)	218.2 (554)
		1800 (980)	33 (227.5)	107.5 (273)
		2000 (1095)	15 (103)	48.8 (124)
		2100 (1150)	9 (62)	29.3 (74)
Alloy 2	0.275 (7.60)	1400 (760)	67 (462)	244.1 (620)
		2000 (1095)	14.5 (100)	52.8 (134)
		2100 (1150)	11 (76)	40.1 (102)
Alloy 8	0.295 (8.15)	1400 (760)	72 (496)	244.1 (620)
		2000 (1095)	18 (124)	61.0 (155)
		2100 (1150)	13.7 (94)	46.4 (118)
Alloy 16	0.274 (7.57)	1400 (760)	72 (496)	263.3 (669)
		2000 (1095)	15.3 (105)	55.9 (142)
		2100 (1150)	12.6 (87)	46.1 (117)
Alloy 23	0.271 (7.49)	1400 (760)	64 (441)	236.5 (601)
		2000 (1095)	12 (83)	44.3 (113)
		2100 (1150)	8 (55)	30 (76)

\*From Reference 10.

TABLE IX

## TENSILE TEST RESULTS

Alloy No.	Bar No.	Zone Anneal Temperature °F (°C)	Zone Anneal Speed lps (cm/s)	Tensile Test Temperature °F (°C)	0.2% PS ksi (MPa)	U.T.S. ksi (MPa)	El. (%)	R.A. (%)	Modulus psi x 10 <sup>6</sup> (MPa x 10 <sup>3</sup> )
8	V178-1	2245 (1230)	3 (7.6)	Room Temp.	112.1 (773)	150.9 (1040)	7.0	13.5	35.9 (247.5)
	V178-1	2245 (1230)	3 (7.6)	1400 (760)	84.4 (582)	122.2 (843)	3.5	11.5	10.3 (71.0)
9	V42A-1	2390 (1310)	5.3 (13.5)	Room Temp.	106.9 (737)	154.5 (1065)	5.5	10.0	37.1 (255.0)
	V42A-3	2410 (1320)	5.3 (13.5)	1400 (760)	112.6 (776)	122.1 (842)	3.5	9.0	10.1 (69.6)
12	V490-3	2355 (1290)	5.3 (13.5)	Room Temp.	121.6 (838)	167.6 (1156)	5.5	13.0	37.8 (260.6)
	V490-2	2355 (1290)	5.3 (13.5)	1400 (760)	114.9 (792)	125.2 (863)	3.5	19.0	10.6 (73.1)
15	V590-1	2290 (1255)	2.8 (7.1)	Room Temp.	115.5 (796)	151.4 (1044)	3.5	10.5	31.8 (219.3)
	V590-1	2290 (1255)	2.8 (7.1)	1400 (760)	114.4 (760)	119.4 (823)	3.5	11.5	7.8 (53.0)
16	V610-1	2315 (1270)*	3.0 (7.6)	Room Temp.	126.4 (871)	143.8 (991)	3.5	6.5	32.8 (226.2)
	V610-1	2315 (1270)*	3.0 (7.6)	1400 (760)	120.4 (830)	131.3 (905)	5.5	0.0	11.0 (81.4)
19	V710-1	2340 (1280)*	3.0 (7.6)	Room Temp.	118.4 (816)	132.6 (914)	3.5	4.5	32.9 (226.0)
	V710-1	2340 (1280)*	3.0 (7.6)	1400 (760)	75.5 (520)	76.8 (530)	<1.0	Nil	9.3 (64.1)
23	V810-1	2360 (1295)*	3.0 (7.6)	Room Temp.	115.2 (794)	130.4 (954)	3.5	6.5	30.6 (211.0)
	V810-1	2360 (1295)*	3.0 (7.6)	1400 (760)	107.6 (742)	120.8 (833)	7.0	10.0	8.3 (57.2)
D.S. Cast Mar. M-200	--	--	--	Room Temp.	120 (827)	135 (930)	7.0	--	31.6 (210.0)
	--	--	--	1400 (760)	122 (841)	135 (930)	3.4	--	25.8 (178.0)
2	V21H	2395 (1300)	5.3 (13.5)	Room Temp.	106.5 (734)	135.8 (916)	3.5	10.0	27.4 (189.0)
	V21H	2395 (1300)	5.3 (13.5)	1400 (760)	95.5 (650)	104.3 (719)	2.0	3.5	10.9 (75.1)

\*Post zone anneal heat treatment given: 1/2 hr/zone anneal temperature/AC.



TABLE X

$\gamma'$  VOLUME FRACTION DETERMINATIONS<sup>+</sup>

<u>Alloy No.</u>	<u>Bar No.</u>	<u>Zone Anneal Temperature °F (°C)</u>	<u>Speed ips (cms)</u>	<u>% <math>\gamma'</math></u>
2	V21C-39	2420 (1327)	5.2 (13.2)	60
8	V37B	2275 (1250)	5.3 (13.5)	60
9	V42A	2380 (1305)	3.0 ( 7.6)	60
12	V49D	2380 (1305)	3.0 ( 7.6)	50
15	V59B	2290 (1255)	2.8 ( 7.1)	60
16	V63B*	2350 (1290)	5.3 (13.5)	65
18	V68D*	2350 (1290)	5.3 (13.5)	65
19	V71D*	2320 (1270)	3.0 ( 7.6)	65
23	V81D	2320 (1270)	2.7 ( 6.9)	65

<sup>+</sup>All samples heat treated 1/2 hr/2000°F(1095°C)/WQ prior to  $\gamma'$  volume fraction determinations.

\*Post zone anneal heat treatment given: 1/2 hr/zone anneal temperature/AC.

TABLE XI  
BURNER RIG SULFIDATION TEST RESULTS<sup>+</sup>

Alloy No.*	Bar No.	Test Duration (hr)	$\Delta W$ Undesc. $10^{-3}$ lb/in <sup>2</sup> (mg/cm <sup>2</sup> )	$\Delta W$ Desc. $10^{-3}$ lb/in <sup>2</sup> (mg/cm <sup>2</sup> )	Metal Loss $10^{-4}$ in ( $\mu$ m)	Maximum Attack $10^{-4}$ in ( $\mu$ m)
2	V21I	168	-3.24 (-228)	-3.99 (-281)	563 (1430)	586 (1488)
	V21I	168	-3.72 (-262)	-4.25 (-299)	569 (1443)	630 (1600)
8	V37B	168	-4.03 (-284)	-7.10 (-500)	N.D.**	N.D.
	V37B	168	-5.01 (-353)	-6.72 (-473)	N.D.	N.D.
	V37B	168	-4.72 (-332)			
9	V42A	168	-1.86 (-131)	-2.65 (-186)	213 (541)	379 (963)
	V42A	168	-2.92 (-205)			
12	V49D	168	-2.23 (-157)	-2.92 (-205)	475 (1207)	1250 (3175)
	V49D	168	-2.05 (-144)			
15	V59B	240	-5.25 (-369)	-5.45 (-383)	348 (884)	406 (1031)
	V59B	240	-4.11 (-289)			
16	V63B	240	-4.84 (-340)	-5.06 (-356)	348 (909)	413 (1049)
	V63B	240	-4.93 (-347)			
17	V66D	168	-3.00 (-211)	-3.57 (-251)	268 (681)	349 (887)
	V66D	168	-2.43 (-171)			

<sup>+</sup> Conditions: 1700°F (930°C) (58 minutes) followed by 2 minute air blast.

\* Compositions - See Table I.

\*\*N.D. = Not Determined.

TABLE XI (CONTINUED)

Alloy No.*	Bar No.	Test Duration (hr)	$\Delta W$ Undesc. $10^{-3}$ lb/in <sup>2</sup> (mg/cm <sup>2</sup> )	$\Delta W$ Desc. $10^{-3}$ lb/in <sup>2</sup> (mg/cm <sup>2</sup> )	Metal Loss $10^{-4}$ in ( $\mu$ m)	Maximum Attack $10^{-4}$ in ( $\mu$ m)
18	V68D	168	-3.34 (-235)	-3.90 (-274)	280 (711)	332 (843)
	V68D	168	-2.76 (-194)			
19	V71D	144	+0.95 (+57)		DESTROYED	
	V71D	144	+1.44 (+101)		DESTROYED	
22	V78D	168	-2.70 (-190)	-3.21 (-226)	363 (922)	456 (1158)
	V78D	168	-3.17 (-223)			
23	V81D	240	-3.28 (-231)	-3.63 (-255)	234 (594)	282 (716)
	V81D	240	-2.77 (-195)			
	V81D	168	-1.75 (-123)			
	V81D	168	-1.53 (-108)			
	V81D	168	-2.32 (-163)	-3.15 (-222)	215 (546)	215 (546)
IN-100	--	96	-6.17 (-434)		DESTROYED	
IN-738	--	240	+0.057 (+4)	-0.057 (-4)	+7 (+18)	308 (782)
IN-713C	--	192	-1.81 (-127)		DESTROYED	

+ Conditions: 1700°F (930°C) (58 minutes) followed by 2 minute air blast.

\* Compositions - See Table I.

\*\*N.D. = Not Determined.

TABLE XII

2012°F (1100°C) CYCLIC OXIDATION TEST RESULTS\*

Alloy No. <sup>†</sup>	Bar No.	Test Duration (hrs)	$\Delta W$ Undesc. $10^{-3}$ lb/in <sup>2</sup> (mg/cm <sup>2</sup> )	$\Delta W$ Desc. $10^{-3}$ lb/in <sup>2</sup> (mg/cm <sup>2</sup> )
2	V21G	504	-0.016 (-1.15)	-0.035 (-2.47)
	V21G	504	-0.014 (-1.00)	-0.033 (-2.30)
8	V37B	504	-0.173 (-12.34)	-0.199 (-14.24)
	V37B	504	-0.177 (-12.67)	
9	V42A	504	-0.039 (-2.78)	-0.057 (-3.98)
	V42A	504	-0.042 (-2.94)	
12	V49D	504	-0.113 (-7.97)	-0.137 (-9.67)
	V49D	504	-0.118 (-8.20)	
15	V59B	504	-0.008 (-0.55)	-0.005 (-0.35)
	V59B	504	-0.010 (-0.68)	
16	V63B	504	-0.007 (-0.46)	-0.016 (-1.13)
	V63B	504	-0.007 (-0.49)	
17	V66D	504	-0.008 (-0.53)	-0.023 (-1.61)
	V66D	504	-0.008 (-0.53)	
18	V68D	504	-0.016 (-1.10)	-0.030 (-2.10)
	V68D	504	-0.012 (-0.82)	
19	V71D	504	-0.131 (+9.18)	-0.109 (+7.64)
	V71D	504	-0.042 (+2.98)	
22	V78D	504	-0.015 (+1.02)	-0.010 (-0.69)
	V78D	504	-0.016 (+1.13)	
23	V81D	504	-0.010 (-0.69)	-0.026 (-1.83)
	V81D	504	-0.012 (-0.81)	
IN-100		504	-0.042 (-2.99)	-0.10 (-7.27)
IN-713LC		504	-0.23 (-16.48)	-0.25 (-17.91)
IN-713C		504	-0.20 (-14.07)	-0.22 (-15.37)
IN-738		504	-0.87 (-61.46)	-1.02 (-71.91)

\*Conditions: Air-5% $H_2O$  flowing at 250 cc/min (15 in /min). Samples cycled to room temperature every 24 hours.<sup>†</sup>See Table I for compositions.



TABLE XIII  
SERIES II ALLOYS (COMPOSITIONS IN ATOMIC AND WEIGHT %)

Alloy No.	Atomic % <sup>a</sup>										Weight %										
	Ni	Cr	Al	Co	Mo	W	Hf	Ta	Nb	Ti	Ni	Cr	Al	Co	Mo	W	Hf	Ta	Nb	Ti	Y <sub>2</sub> O <sub>3</sub>
V86-25	Bal.	15.0	16.5	10.0	2.0	--	--	1.0	--	--	Bal.	14.3	0.2	10.0	3.5	--	--	3.3	--	--	1.1
V89-26	Bal.	15.0	15.5	5.0	--	2.0	--	1.0	1.0	--	Bal.	13.7	7.4	5.2	--	6.5	--	3.2	1.6	--	1.1
V92-27	Bal.	15.0	16.5	10.0	2.0	2.0	--	--	1.0	--	Bal.	13.9	7.9	10.5	3.4	6.6	--	--	1.7	--	1.1
V95-28	Bal.	15.0	14.5	5.0	--	--	--	1.0	1.0	--	Bal.	14.0	7.0	5.3	--	--	3.2	3.2	1.7	--	1.1
V97-29	Bal.	15.0	15.0	5.0	--	--	--	0.5	1.0	--	Bal.	14.2	7.3	5.3	--	--	1.6	3.3	1.7	--	1.1
V99-30	Bal.	15.0	16.0	10.0	2.0	--	--	0.5	--	1.0	Bal.	14.2	7.9	10.7	3.5	--	1.6	--	1.7	--	1.1
V109-31	Bal.	15.0	17.0	10.0	--	2.0	0.5	--	--	--	Bal.	14.1	0.3	10.6	--	6.6	1.6	--	--	--	1.1
V102-32	Bal.	15.0	16.0	5.0	2.0	2.0	0.5	1.0	--	--	Bal.	13.5	7.5	5.1	3.3	6.4	1.5	3.1	--	--	1.1
V106-33	Bal.	15.0	16.5	10.0	2.0	2.0	--	--	--	1.0	Bal.	14.0	0.0	10.6	3.4	6.6	--	--	--	0.9	1.1
V114-34	Bal.	15.0	17.5	--	2.0	2.0	--	--	--	--	Bal.	14.1	8.5	--	3.5	6.6	--	--	--	--	1.1
V118-35	Bal.	15.0	17.0	--	1.0	2.0	--	0.5	--	--	Bal.	14.0	8.2	--	1.7	6.6	--	1.6	--	--	1.1
V116-36	Bal.	15.0	16.5	--	--	2.0	--	1.0	--	--	Bal.	13.9	7.9	--	--	6.5	--	3.2	--	--	1.1

<sup>a</sup>Excluded Hf added Y<sub>2</sub>O<sub>3</sub>.

TABLE XIV

## POWDER CHARACTERIZATION OF SERIES II ALLOYS

Alloy No.	Batch No.	Chemistry, Wt. %		Screen Analysis, Mesh Size, %									
		O	N	C	+40	-40/ +80	+100	-80/ +140	-100/ +200	-140/ +230	-200/ +325	-230/ +325	
25	V-86*	.13	.056	.058	15.1	34.6	6.8	8.4	10.1	4.4	7.4	13.2	
	V-87	.12	.07	.54	4.9	18.2	5.2	10.1	16.2	8.0	10.5	26.9	
26	V-89	.18	.07	.060	10.6	27.3	7.4	11.0	14.1	5.6	8.0	16.0	
	V-90	.39	.066	.058	9.8	26.7	7.4	10.6	14.2	6.1	9.3	15.9	
27	V-92	.43	.07	.056	13.4	33.7	9.8	10.6	10.2	4.4	7.0	10.9	
	V-93	.039	.07	.053	9.8	29.3	7.8	10.4	12.0	4.9	8.0	17.8	
28	V-95	.021	.056	.061	13.0	28.1	5.9	11.3	14.0	5.4	8.0	14.3	
	V-96	.19	.052	.061	10.0	27.7	6.1	10.9	13.7	5.9	10.0	15.7	
29	V-97	.28	.060	.061	17.6	33.3	7.5	11.0	12.9	4.3	5.9	7.5	
	V-98	.23	.054	.061	19.6	28.8	6.3	10.6	12.9	3.8	7.2	10.8	
30	V-99	.25	.057	.057	8.3	22.6	6.4	11.7	15.1	5.1	10.3	20.5	
	V-100	.32	.072	.059	1.4	9.3	3.0	7.0	11.9	7.6	13.9	45.9	
31	V-109	.16	.045	.053	10.3	28.5	8.5	12.4	13.5	5.0	9.5	12.3	
	V-110	.25	.048	.052	10.5	30.3	9.0	11.9	13.0	4.2	8.6	12.5	
32	V-102	.24	.062	.054	6.6	17.2	5.0	9.8	15.8	6.5	12.7	26.4	
	V-103	.33	.048	.056	5.1	21.6	6.8	10.9	14.1	6.8	11.8	23.4	
	V-104	.31	.047	.054	8.3	26.2	7.0	10.3	13.4	4.5	10.7	19.6	
33	V-106	.15	.052	.053	9.5	27.6	7.0	10.2	12.5	5.0	9.7	18.5	
	V-107	.18	.051	.052	10.3	26.3	7.0	11.1	13.4	4.8	9.8	17.3	
34	V-114	.40	.059	.081	18.5	33.6	6.6	10.1	10.7	3.6	6.3	10.6	
	V-115	.15	.060	.063	14.4	26.9	6.9	9.8	13.2	2.2	9.8	16.8	
35	V-118	.14	.062	.054	18.0	28.8	7.8	10.7	11.9	2.0	7.2	13.6	
	V-119	.20	.052	.053	13.6	30.6	7.3	11.6	13.5	3.3	7.3	12.8	
36	V-116	.21	.051	.056	21.0	31.7	7.8	10.0	9.9	3.3	6.8	9.5	
	V-117	.27	.059	.055	17.6	31.1	7.7	11.7	12.0	3.4	6.4	10.1	

\*Batch number assignment as indicated in Table II.

TABLE XV

## EXTRUSION CONDITIONS OF SERIES II ALLOYS

Alloy No.	Billet No.	Temperature		Ratio	Ram Speed**	
		°F	(°C)		in/sec	cm/sec
25	V-86A	2150	(1175)	18	5.0	12.7
	V-86B	2100	(1150)	18	5.0	12.7
	V-86C	2050	(1120)	18	5.0	12.7
	V-86D	2100	(1150)	18	15.1	38.4*
26	V-89A	2150	(1175)	18	5.6	14.2
	V-89B	2100	(1150)	18	5.4	13.7
	V-89C	2050	(1120)	18	4.8	12.2
	V-89D	2100	(1150)	18	14.5	36.8*
	V-89E	1850	(1010)	18	3.4	8.6
	V-89F	1950	(1065)	18	3.4	8.6
	V-89G	1850	(1010)	18	7.6	19.3*
27	V-92A	2150	(1175)	18	5.8	14.7
	V-92B	2100	(1150)	18	5.4	13.7
	V-92C	2050	(1120)	18	4.4	11.2
	V-92D	2100	(1150)	18	14.1	35.8*
	V-92E	1950	(1065)	18	3.0	7.6
	V-92F	1850	(1010)	18	12.1	30.7*
	V-92G	1850	(1010)	18	2.4	6.1
28	V-95A	1950	(1065)	18	3.0	7.6
	V-95B	1950	(1065)	18	No Record*	
	V-95C	1850	(1010)	18	3.6	9.1
	V-95D	2050	(1120)	18	No Record*	
29	V-97A	1950	(1065)	18	3.6	9.1
	V-97B	1950	(1065)	18	No Record*	
	V-97C	1850	(1010)	18	1.0	2.5
	V-97D	2050	(1120)	18	No Record	
30	V-99A	1950	(1065)	18	4.0	10.2
	V-99B	1950	(1065)	18	No Record*	
	V-99C	1850	(1010)	18	1.0	2.5
	V-99D	2050	(1120)	18	No Record	

TABLE XV (CONTINUED)

Alloy No.	Billet No.	Temperature °F (°C)	Ratio	Ram Speed**	
				in/sec	cm/sec
32	V-102A	2100 (1150)	18	4.2	10.7
	V-102B	2050 (1120)	30	2.4	6.1
	V-102C	2050 (1120)	18	4.0	10.2
	V-102D	1950 (1065)	18	3.0	7.6
	V-102E	1850 (1010)	18	3.0	7.6
	V-102F	1850 (1010)	18	7.6	19.3
	V-102G	1950 (1065)	18	8.1	20.6*
33	V-106A	2100 (1150)	18	4.2	10.7
	V-106B	2050 (1120)	30	3.6	9.1
	V-106C	2050 (1120)	18	4.0	10.2
	V-106D	1950 (1065)	18	4.4	11.2
	V-106E	1850 (1010)	18	1.0	2.5
	V-106F	1850 (1010)	18	5.8	14.7*
34	V-114A	1850 (1010)	18	0.40	1.0
	V-114B	1950 (1065)	18	1.6	4.1
	V-114C	2050 (1120)	18	8.0	20.3
	V-114D	2100 (1150)	18	No Record	
	V-114E	2050 (1120)	18	14.1	35.8*
35	V-118A	1850 (1010)	18	2.0	5.1
	V-118B	1950 (1065)	18	No Record	
	V-118C	2050 (1120)	18	No Record	
	V-118D	2100 (1150)	18	No Record	
	V-118E	2050 (1120)	18	9.6	23.4*
36	V-116A	1850 (1010)	18	0.81	2.0
	V-116B	1950 (1065)	18	1.6	4.1
	V-116C	2050 (1120)	18	3.6	9.1
	V-116D	2100 (1150)	18	No Record	
	V-116E	2050 (1120)	18	13.7	34.8*

\*100% press throttle

\*\* 35% press throttle



TABLE XV (CONTINUED)

Alloy No.	Billet No.	Temperature °F (°C)	Ratio	Ram Speed**	
				in/sec	cm/sec
32	V-102A	2100 (1150)	18	4.2	10.7
	V-102B	2050 (1120)	30	2.4	6.1
	V-102C	2050 (1120)	18	4.0	10.2
	V-102D	1950 (1065)	18	3.0	7.6
	V-102E	1850 (1010)	18	3.0	7.6
	V-102F	1850 (1010)	18	7.6	19.3
	V-102G	1950 (1065)	18	8.1	20.6*
33	V-106A	2100 (1150)	18	4.2	10.7
	V-106B	2050 (1120)	30	3.6	9.1
	V-106C	2050 (1120)	18	4.0	10.2
	V-106D	1950 (1065)	18	4.4	11.2
	V-106E	1850 (1010)	18	1.0	2.5
	V-106F	1850 (1010)	18	5.8	14.7*
34	V-114A	1850 (1010)	18	0.40	1.0
	V-114B	1950 (1065)	18	1.6	4.1
	V-114C	2050 (1120)	18	8.0	20.3
	V-114D	2100 (1150)	18	No Record	
	V-114E	2050 (1120)	18	14.1	35.8*
35	V-118A	1850 (1010)	18	2.0	5.1
	V-118B	1950 (1065)	18	No Record	
	V-118C	2050 (1120)	18	No Record	
	V-118D	2100 (1150)	18	No Record	
	V-118E	2050 (1120)	18	9.6	23.4*
36	V-116A	1850 (1010)	18	0.81	2.0
	V-116B	1950 (1065)	18	1.6	4.1
	V-116C	2050 (1120)	18	3.6	9.1
	V-116D	2100 (1150)	18	No Record	
	V-116E	2050 (1120)	18	13.7	34.8*

\*100% press throttle

\*\* 35% press throttle



P.N. 1-58819

50X

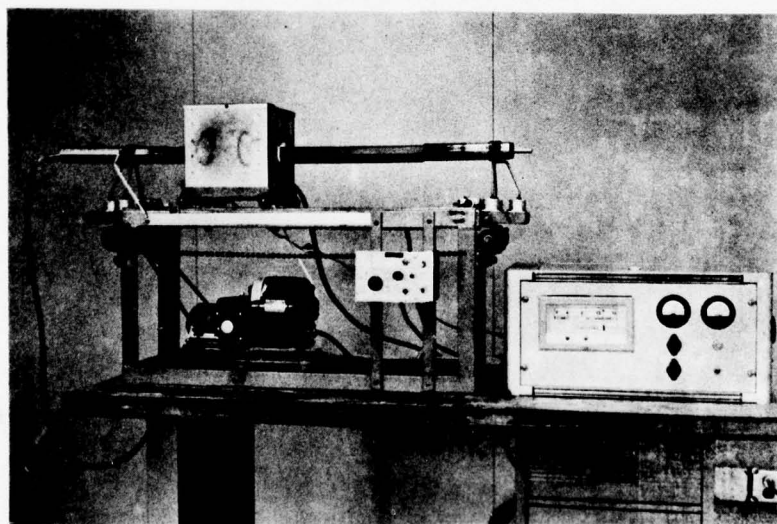
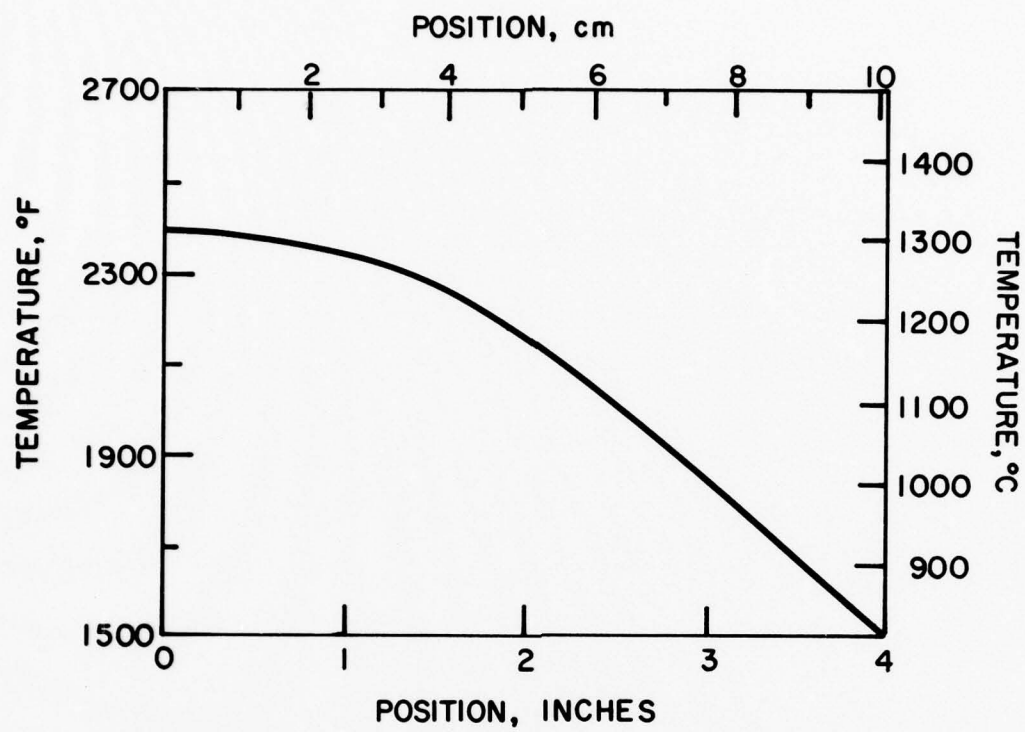


P.N. 1-58820

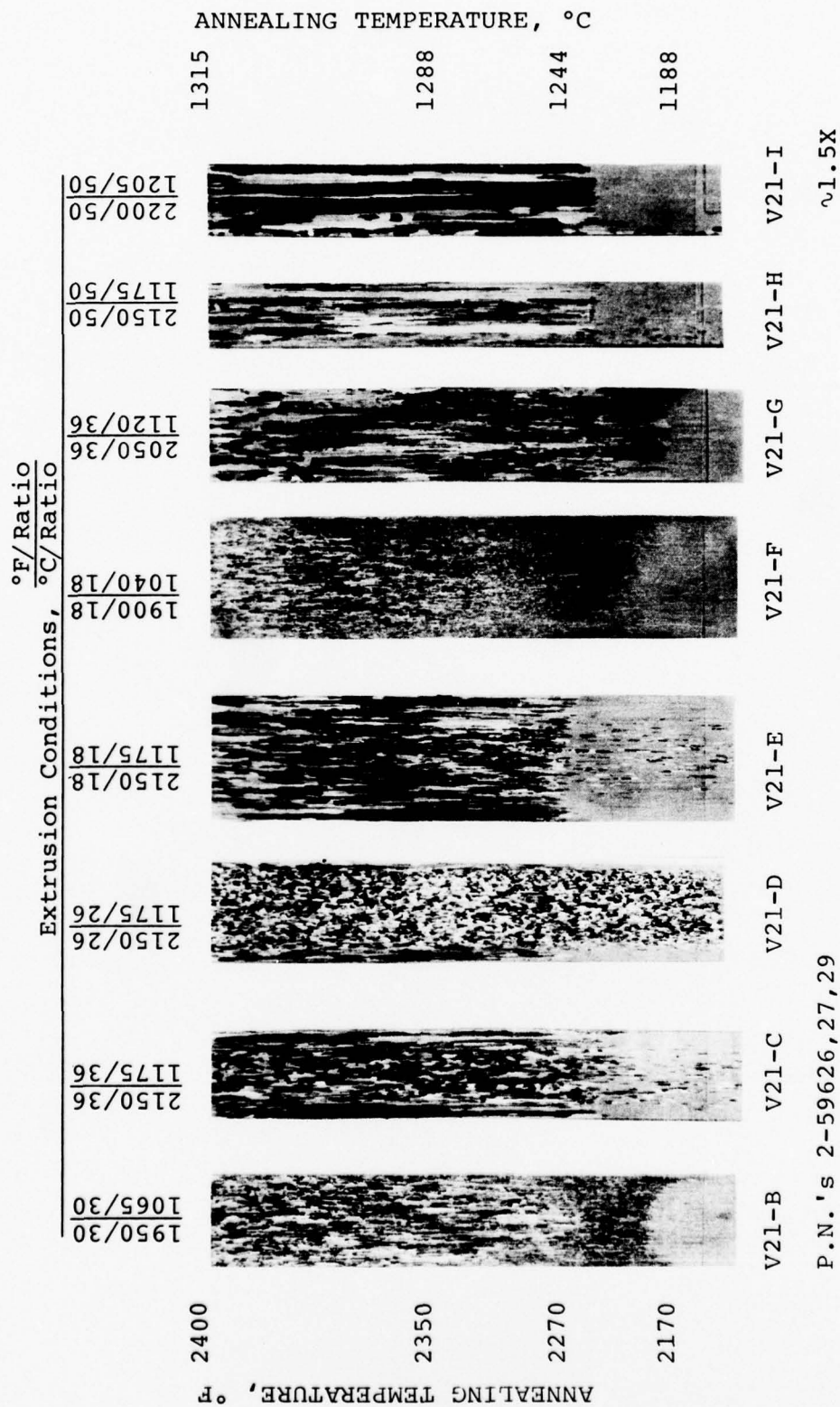
250X

FIGURE 1

MICROGRAPHS OF ATTRITED POWDER OF  
ALLOY 9 (1 AT.% Ta)



**FIGURE 2** - Temperature Profile and Furnace  
Used for Gradient and Zone  
Annealing



**FIGURE 3**  
 MACROGRAPHS OF ALLOY 2 EXTRUDED AND GRADIENT ANNEALED BARS  
 ETCHANT: 45:45:10 HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>



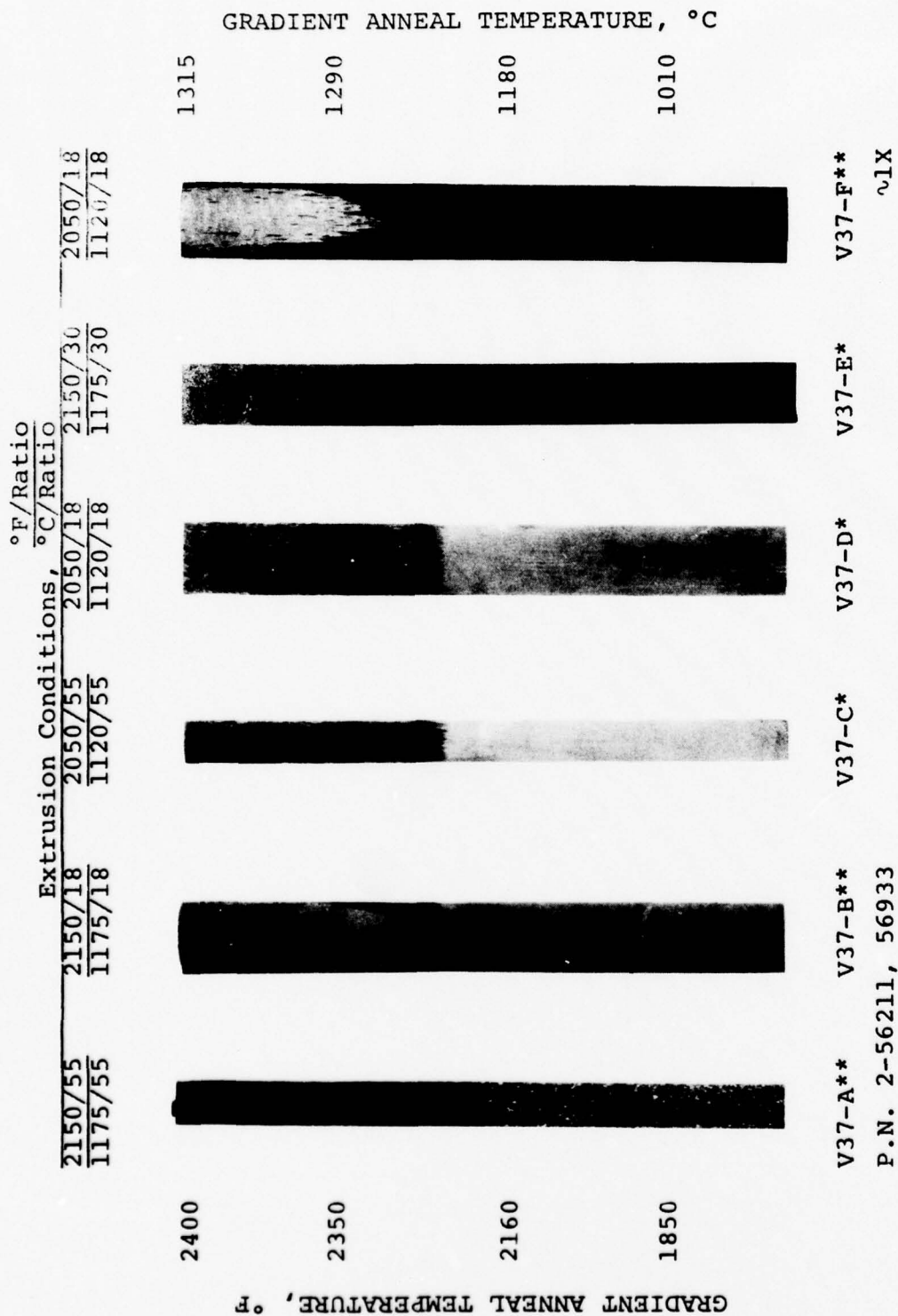
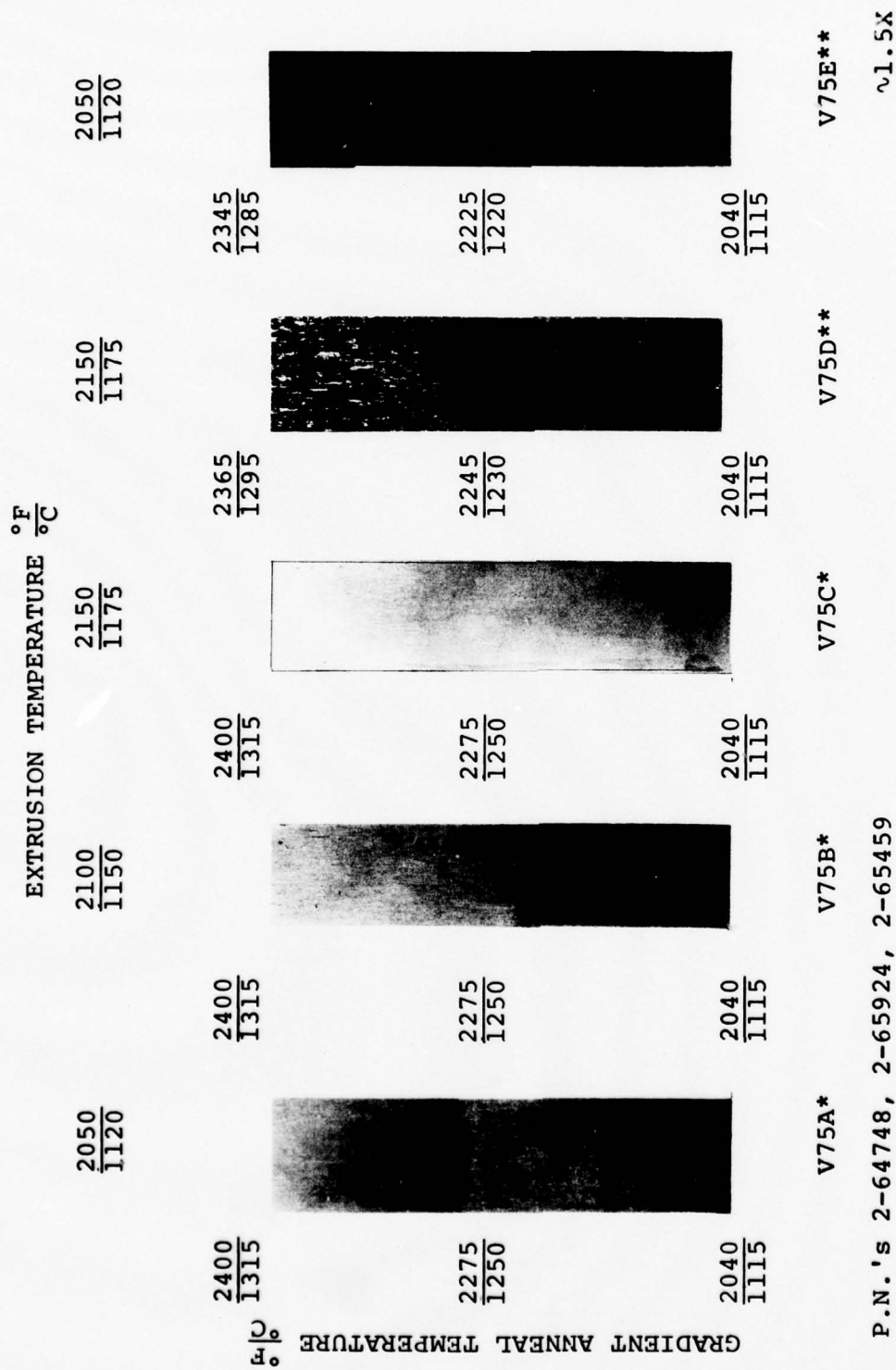


FIGURE 4

MACROGRAPHS OF ALLOY 8 (2 AT.%W) EXTRUDED (\*100%/\*\*35% PRESS THROTTLE) AND GRADIENT ANNEALED BARS

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>

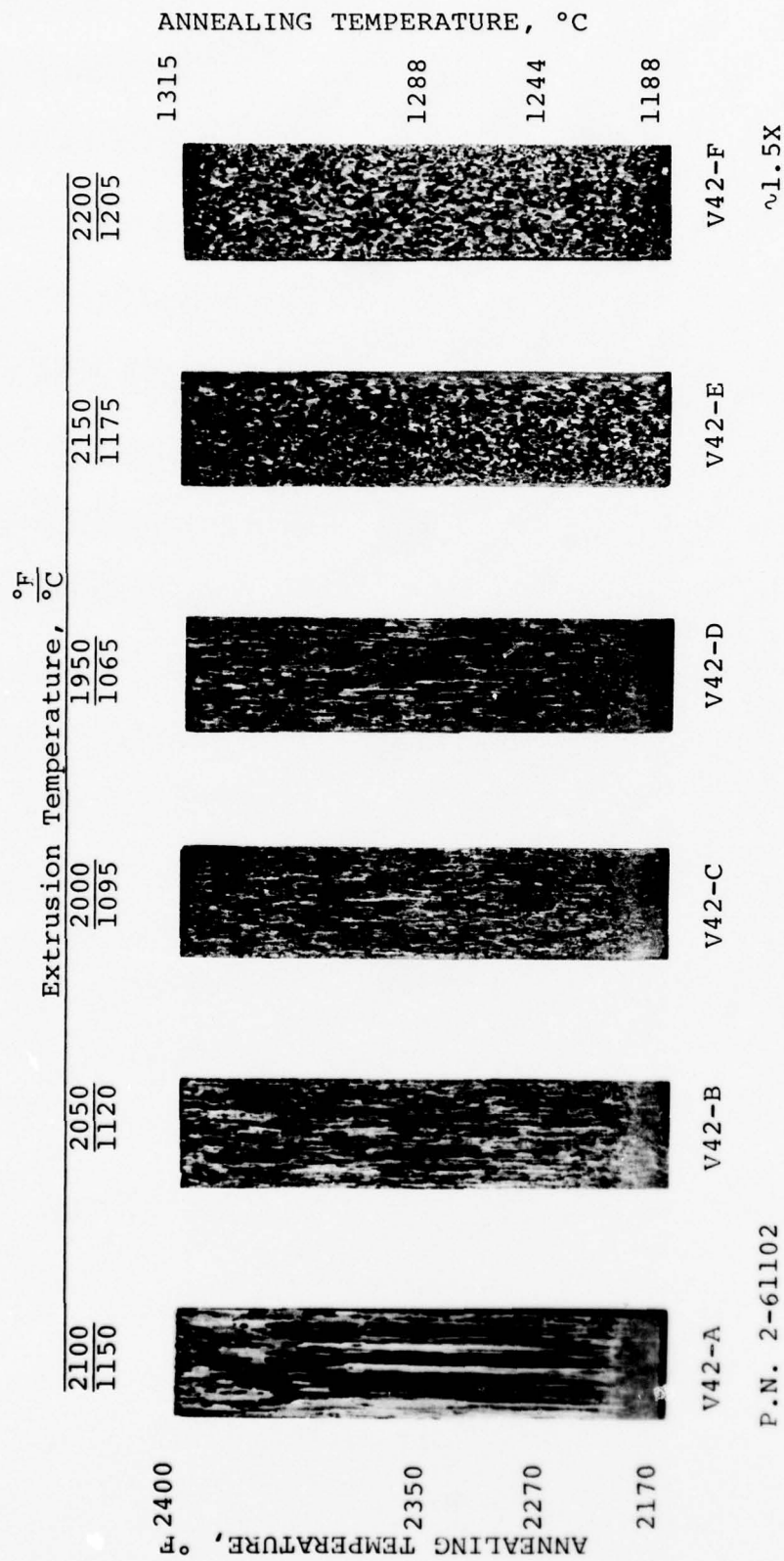


P.N.'s 2-64748, 2-65924, 2-65459

FIGURE 5

MACROGRAPHS OF ALLOY 21 (4 AT. % W)  
EXTRUDED (\*100%/\*\*35% PRESS THROTTLE, 18:1 RATIO)  
AND GRADIENT ANNEALED BARS

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>

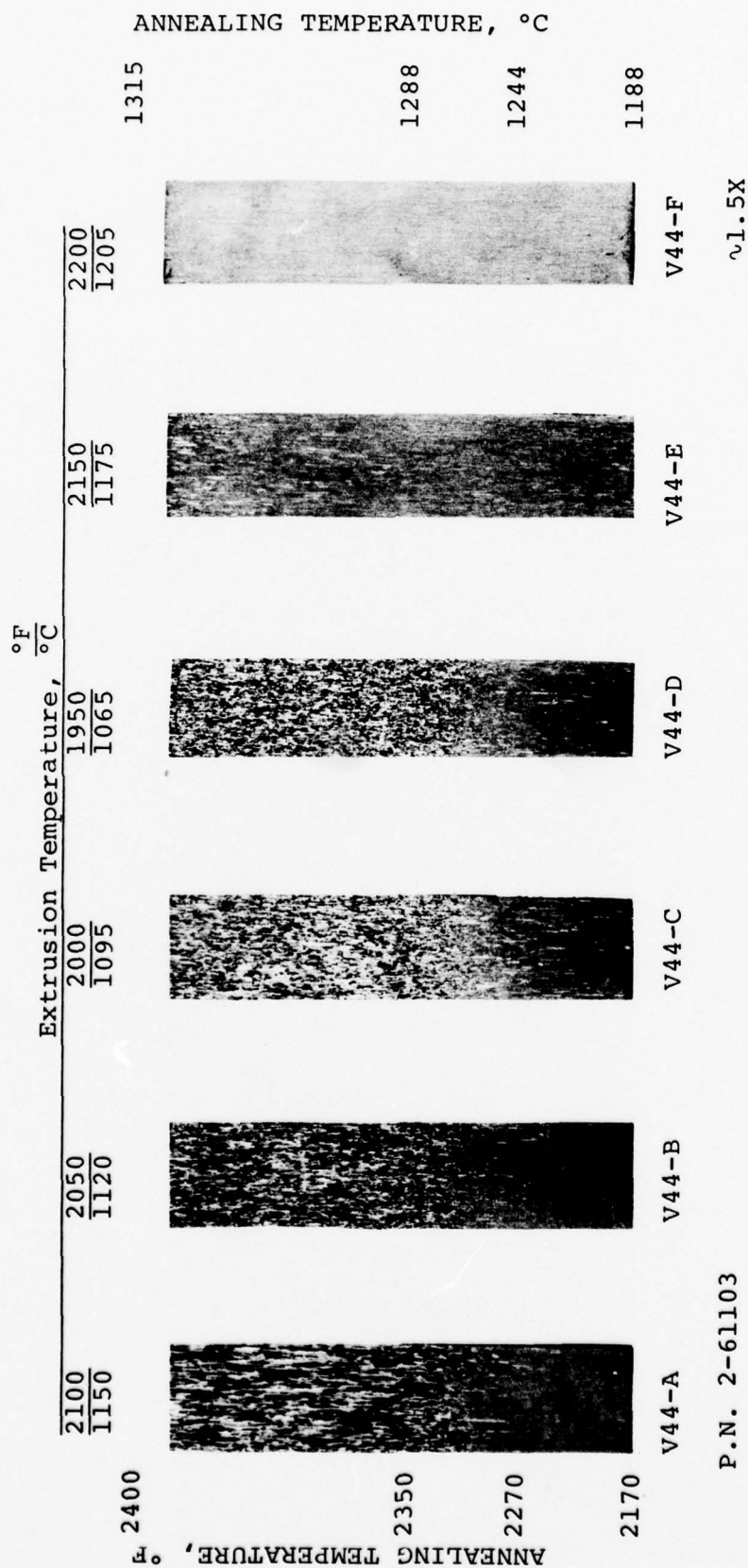


P.N. 2-61102

**FIGURE 6**

MACROGRAPHS OF ALLOY 9 (1 AT. % Ta) EXTRUDED AND GRADIENT ANNEALED BARS

ETCHANT: 45:45:10 HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>



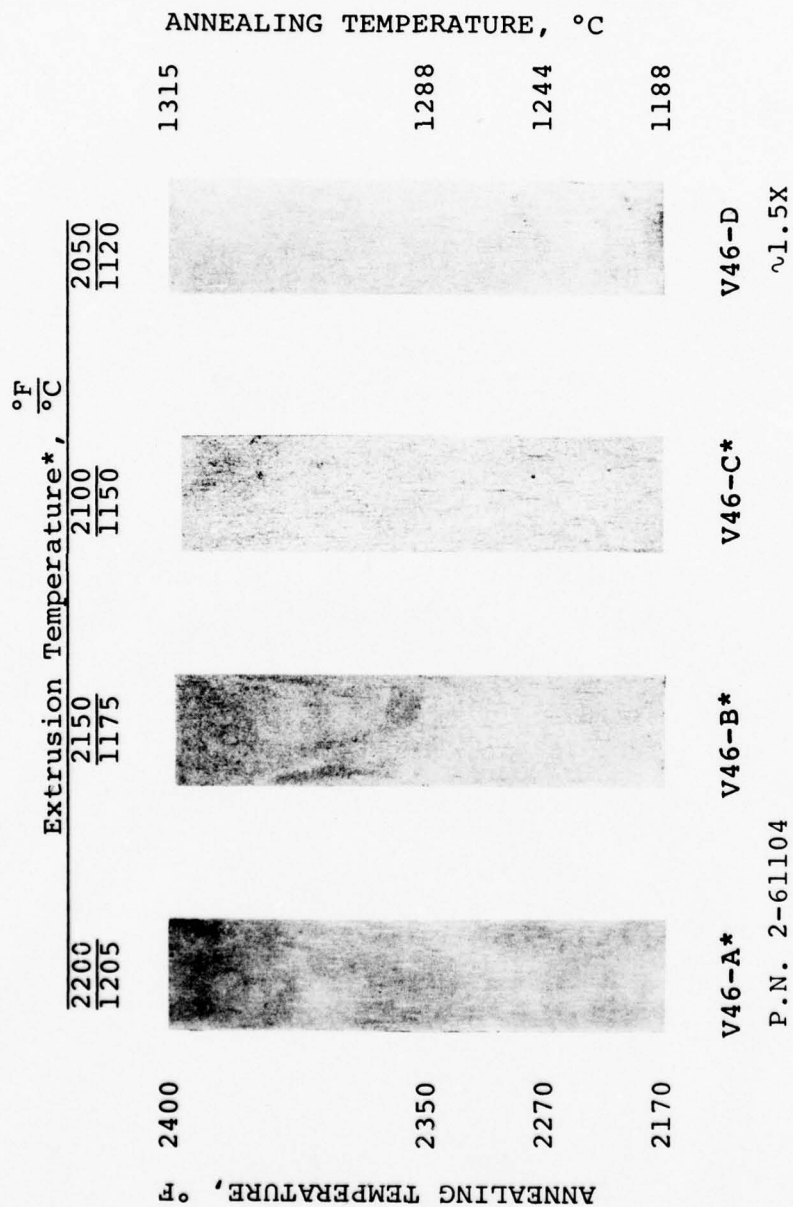
**FIGURE 7**

MACROGRAPHS OF ALLOY 10 (3 AT. % Ta) EXTRUDED AND GRADIENT ANNEALED BARS

ETCHANT: 45:45:10 HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>

P.N. 2-61103





**FIGURE 8**

MACROGRAPHS OF ALLOY 11 (6 AT. % Ta) EXTRUDED  
(\*18:1 RATIO) AND GRADIENT ANNEALED BARS

ETCHANT: 45:45:10 HCL:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>

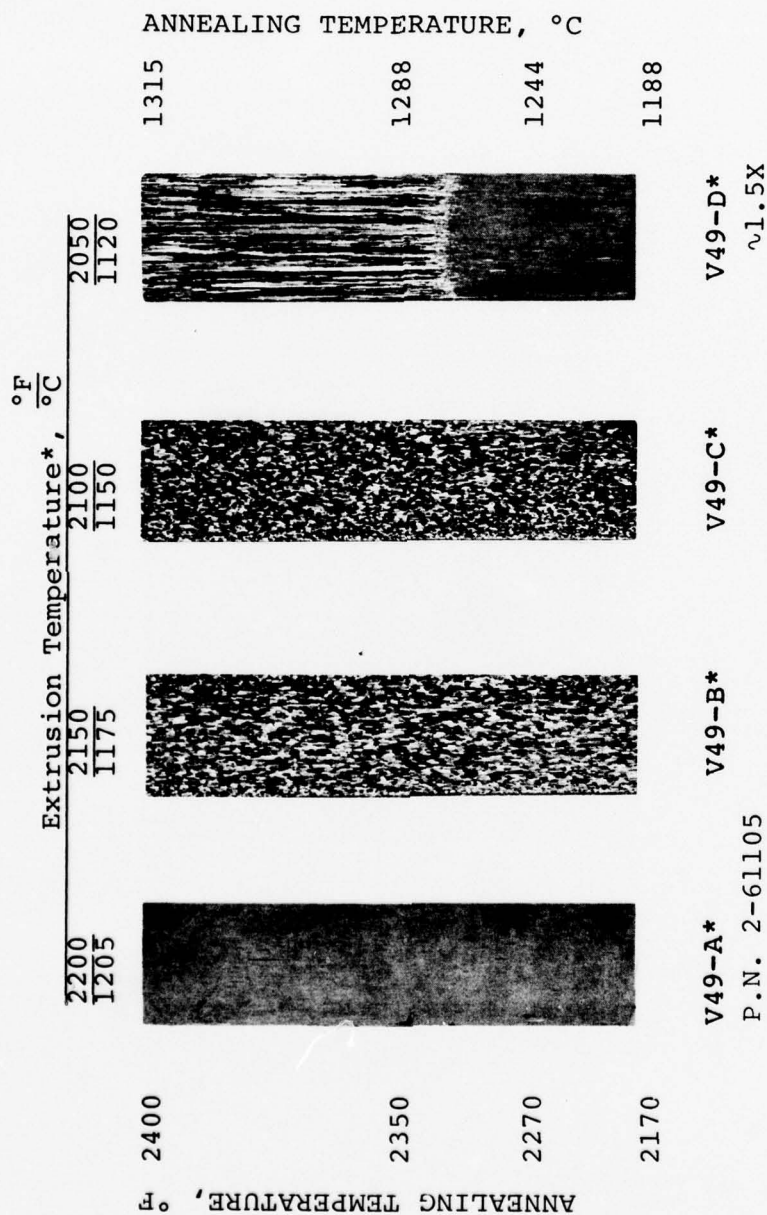
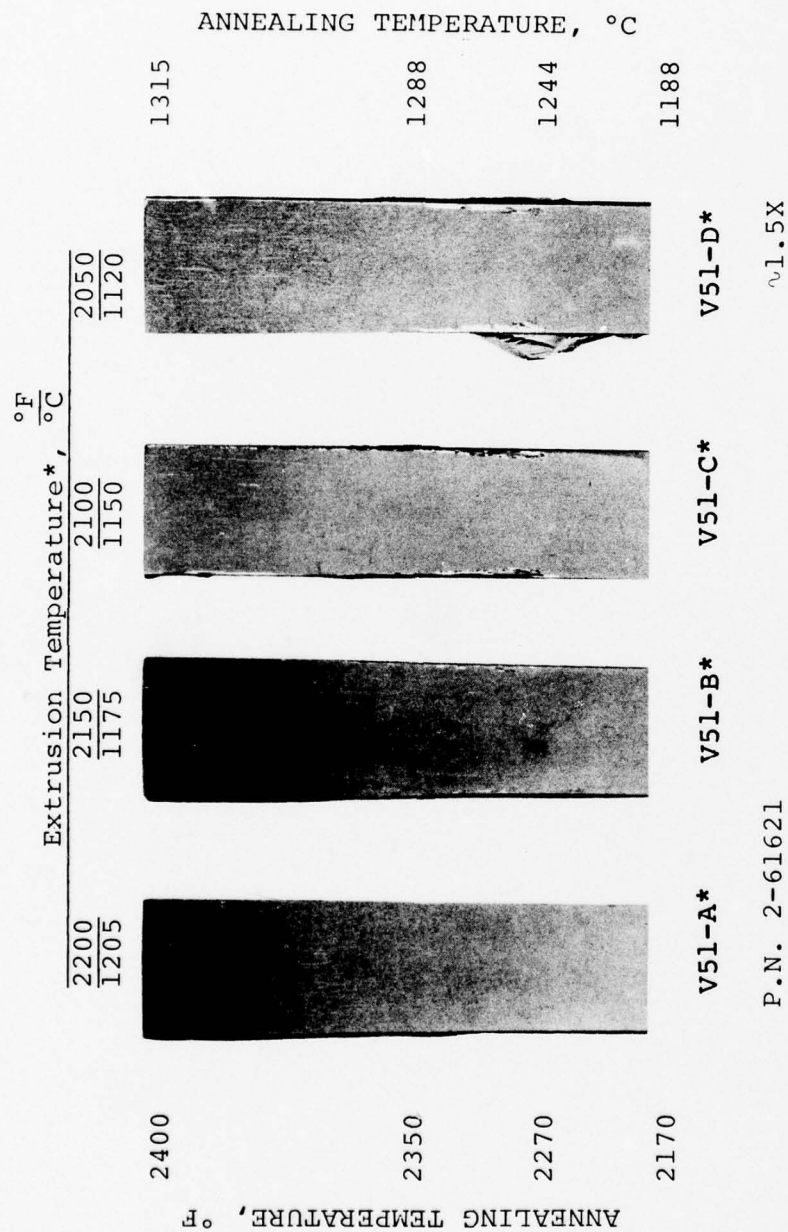


FIGURE 9

MACROGRAPHS OF ALLOY 12 (1 AT.% Nb) EXTRUDED  
(\*18:1 RATIO) AND GRADIENT ANNEALED BARS

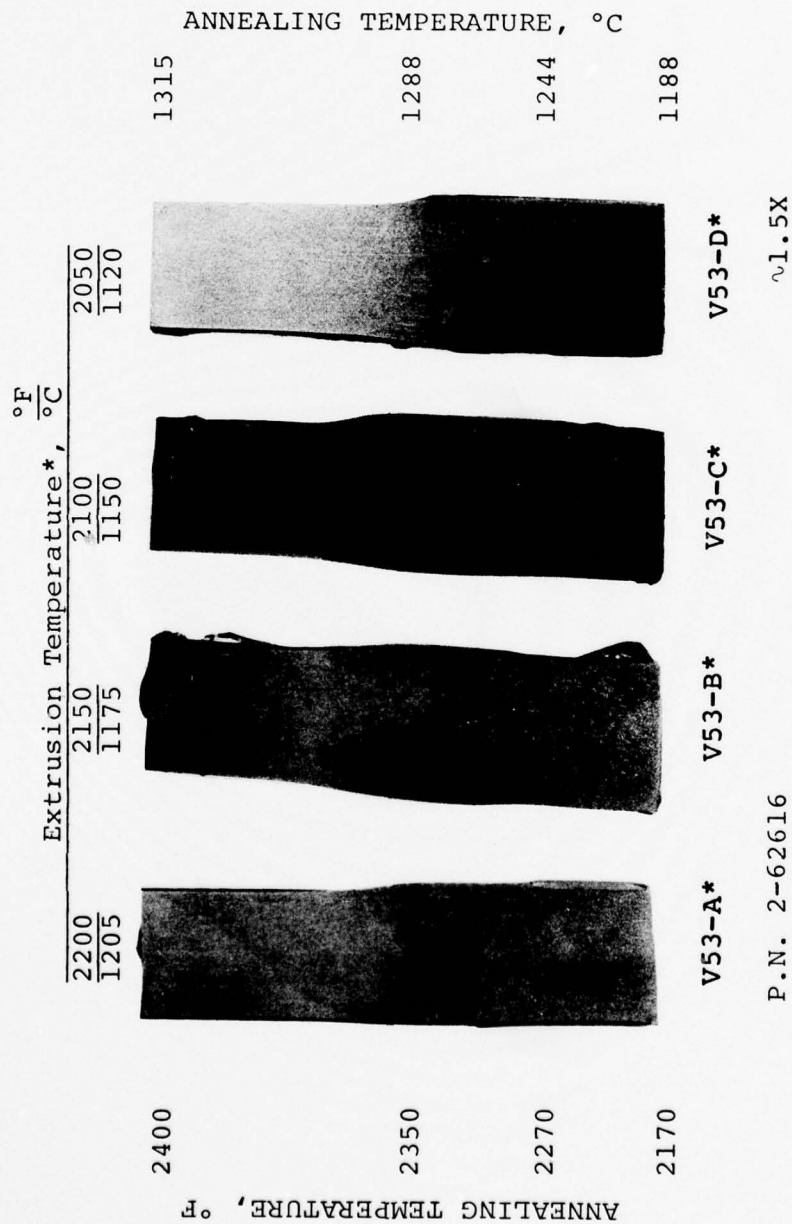
ETCHANT: 45:45:10 HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>



**FIGURE 10**

MICROGRAPHS OF ALLOY 13 (3 AT.% Nb) EXTRUDED  
(\*18:1 RATIO) AND GRADIENT ANNEALED BARS

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>

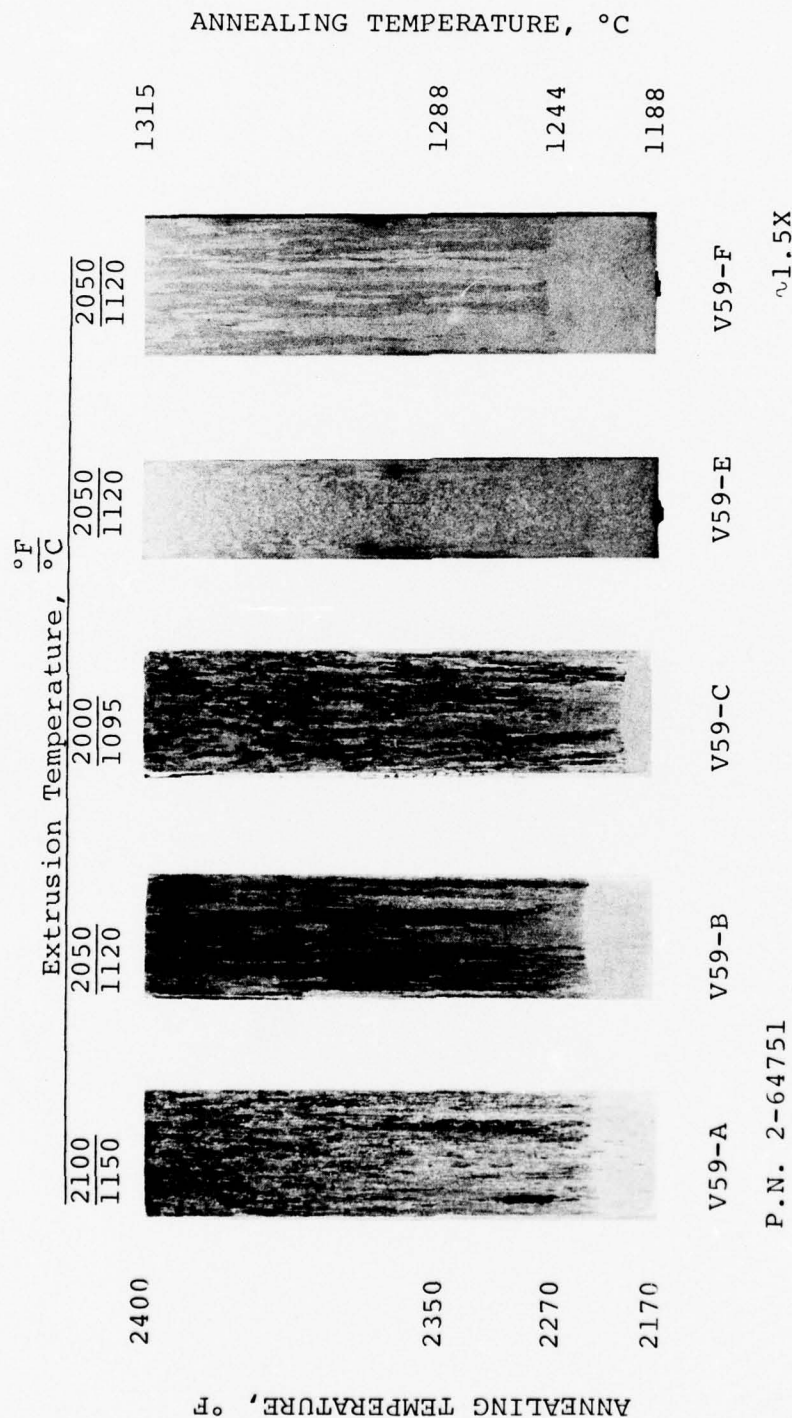


**FIGURE 11**

MICROGRAPHS OF ALLOY 14 (6 AT.% Nb) EXTRUDED  
(\*18:1 RATIO) AND GRADIENT ANNEALED BARS

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>

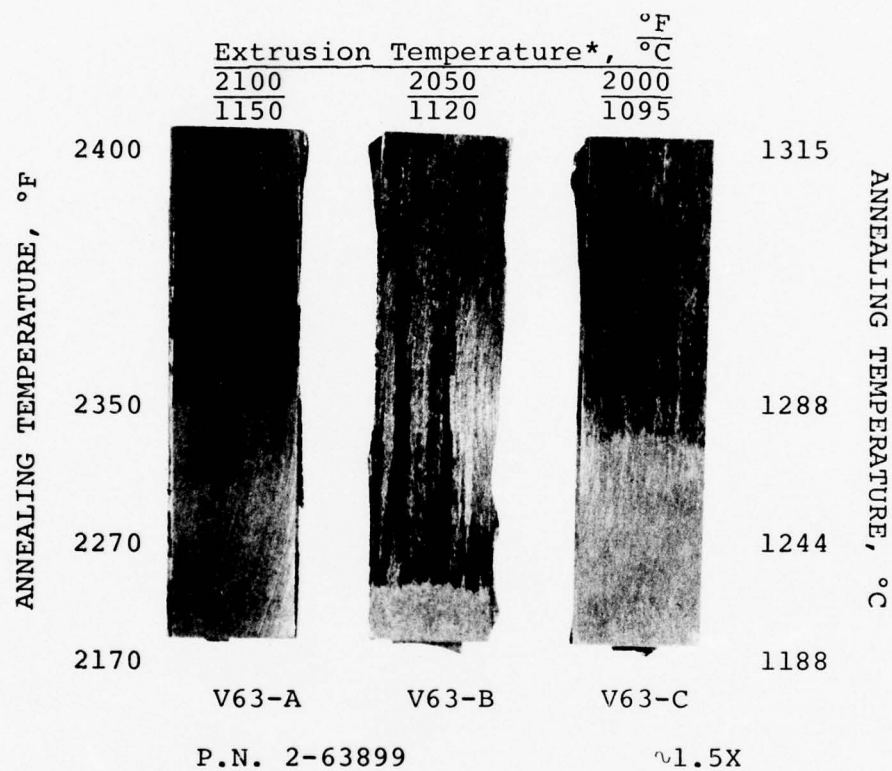




**FIGURE 12**

MICROGRAPHS OF ALLOY 15 (1 AT.% Mo) GRADIENT ANNEALED BAR  
 59-A, 59-B, 59-C EXTRUDED 18:1; 59-E EXTRUDED 36:1;  
 59-F EXTRUDED 16:1

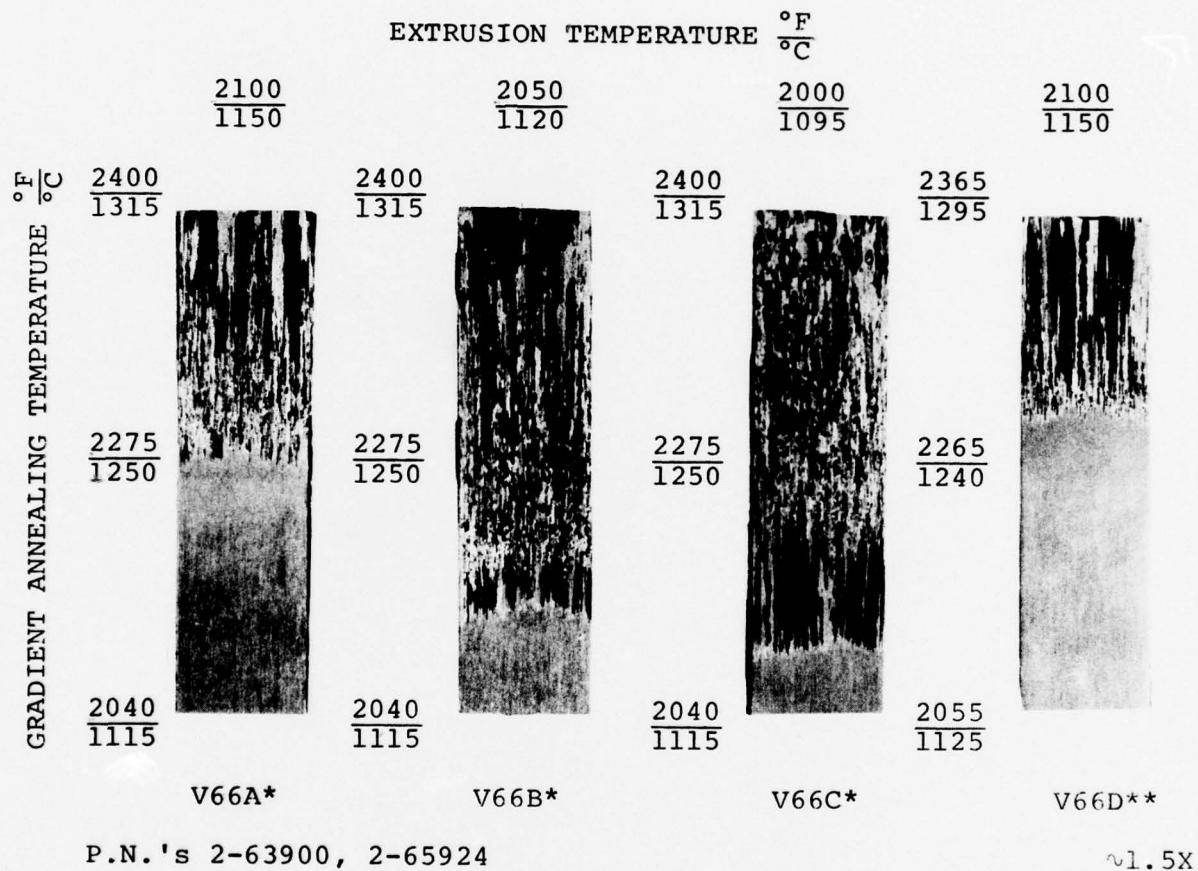
ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>



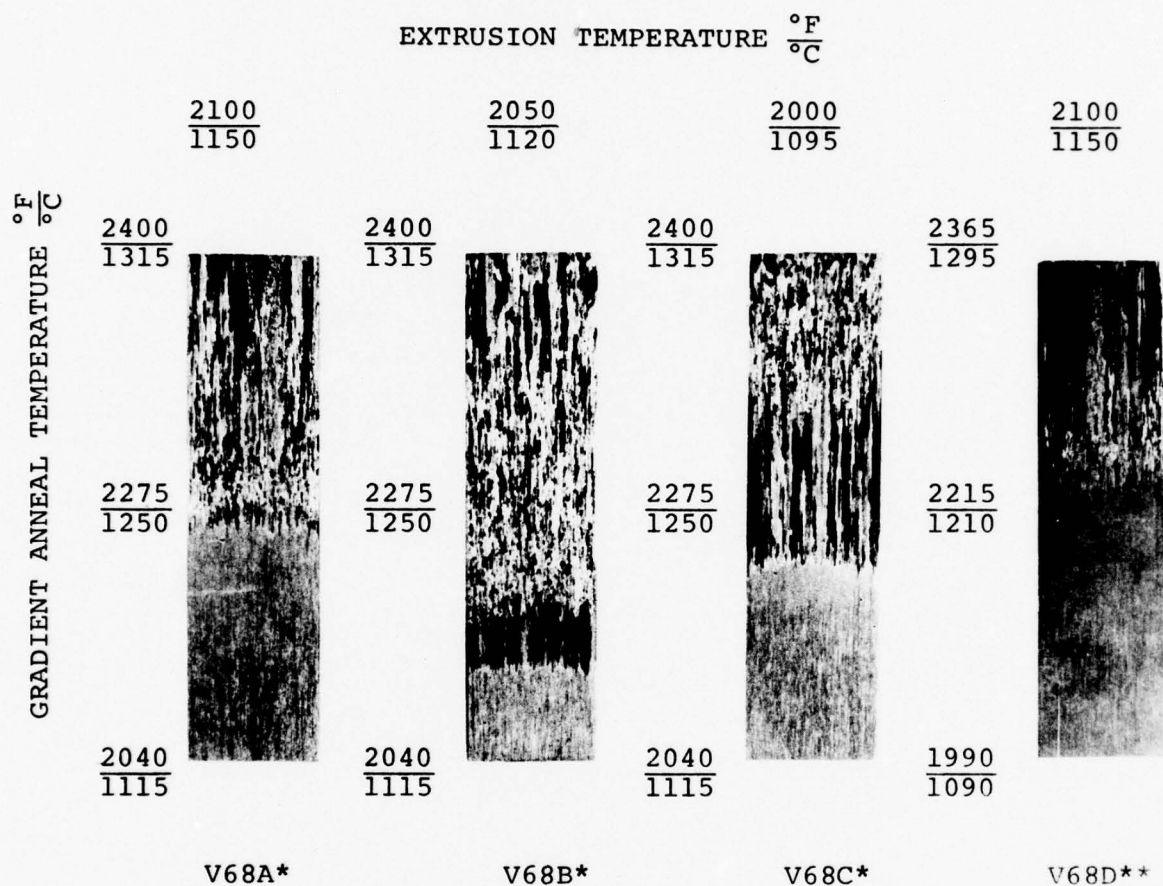
**FIGURE 13**

MICROGRAPHS OF ALLOY 16  
(2 AT.% Mo) EXTRUDED  
(\*18:1 RATIO) AND GRADIENT  
ANNEALED BARS

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>



**FIGURE 14**  
 MACROGRAPHS OF ALLOY 17 (5 AT. % CO)  
 EXTRUDED (\*100%/\*\*35% PRESS THROTTLE, 18:1 RATIO)  
 AND GRADIENT ANNEALED BARS  
 ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>



P.N.'s 2-63567, 2-65924

~1.5X

# **FIGURE 15**

MACROGRAPHS OF ALLOY 18 (10 AT. % CO)  
EXTRUDED (\*100%/\*\*35% PRESS THROTTLE, 18:1 RATIO)  
AND GRADIENT ANNEALED BARS

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>





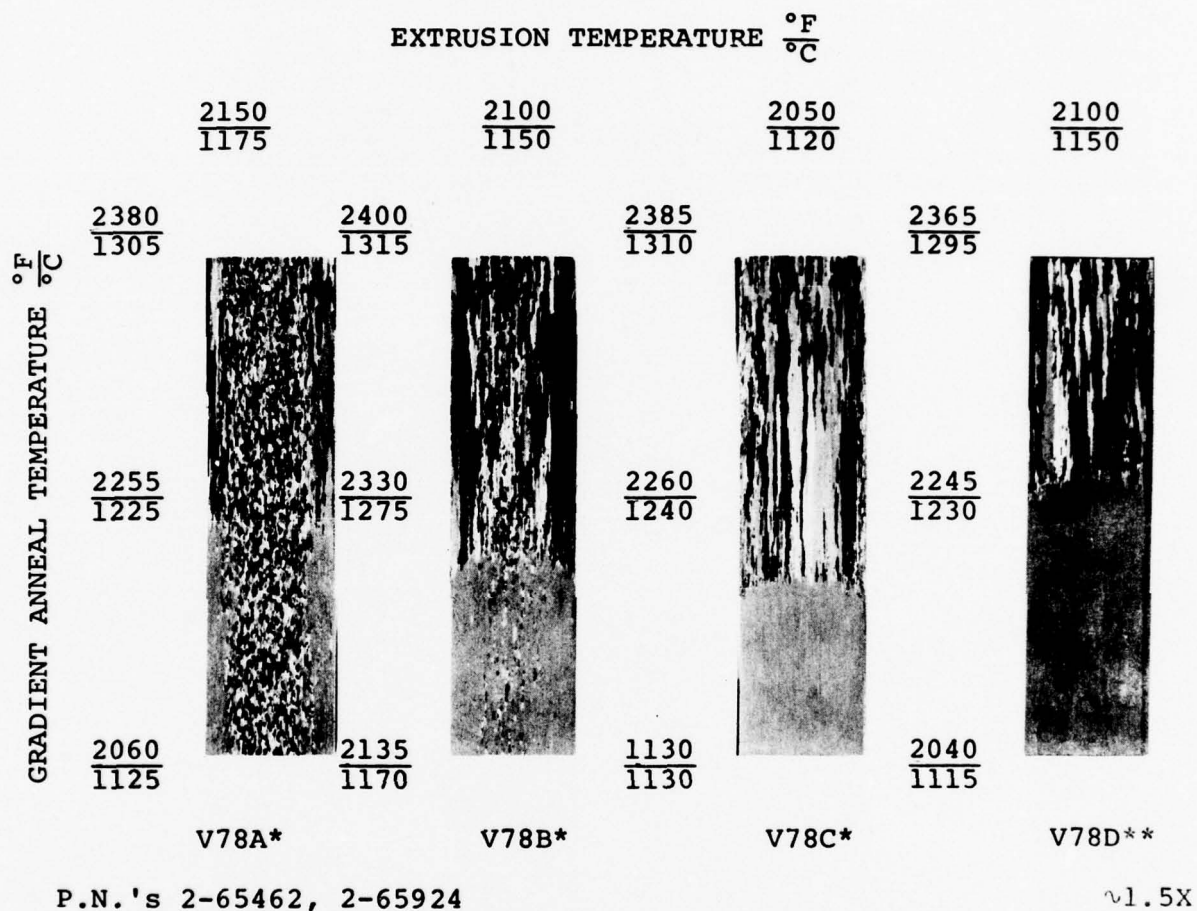


FIGURE 17

MACROGRAPHS OF ALLOY 22 (1 AT. % Ti)  
EXTRUDED (\*100%/\*\*35% PRESS THROTTLE, 18:1 RATIO)  
AND GRADIENT ANNEALED BARS

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>

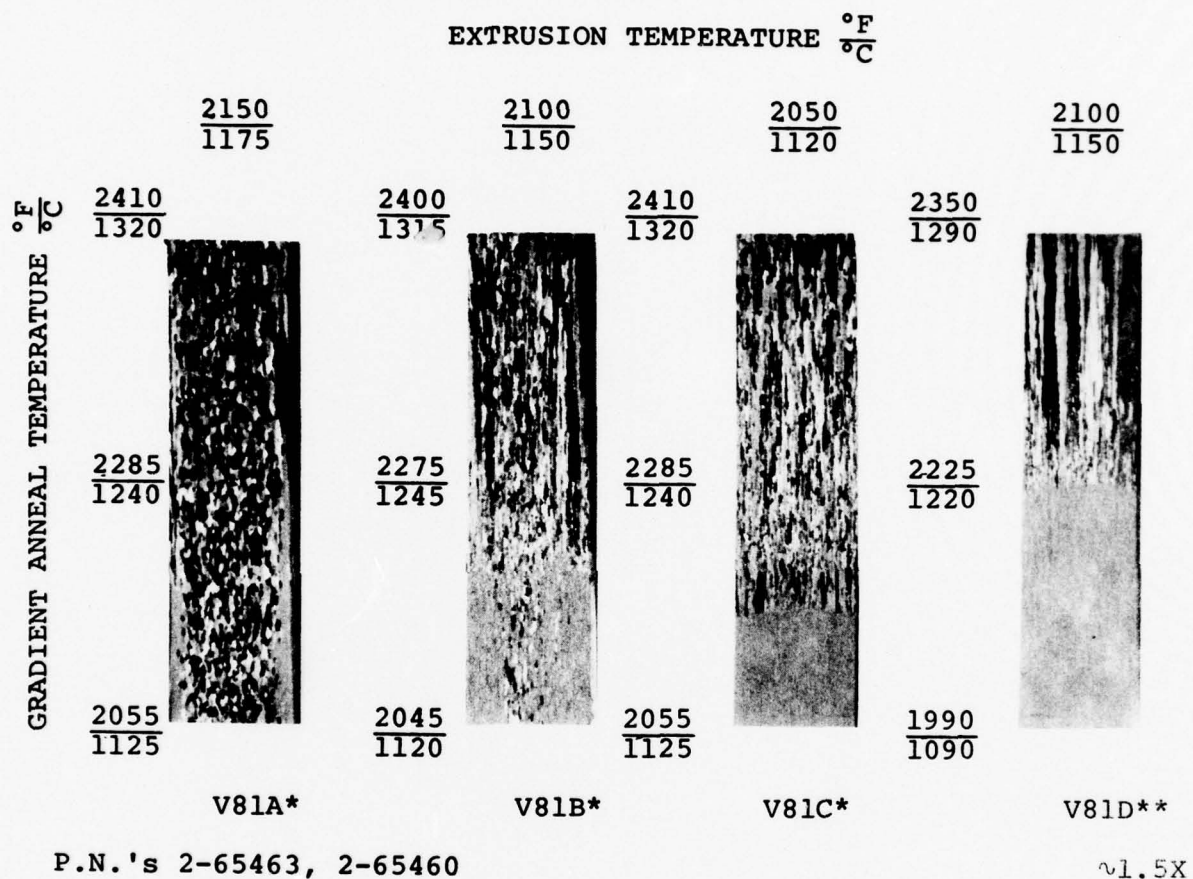
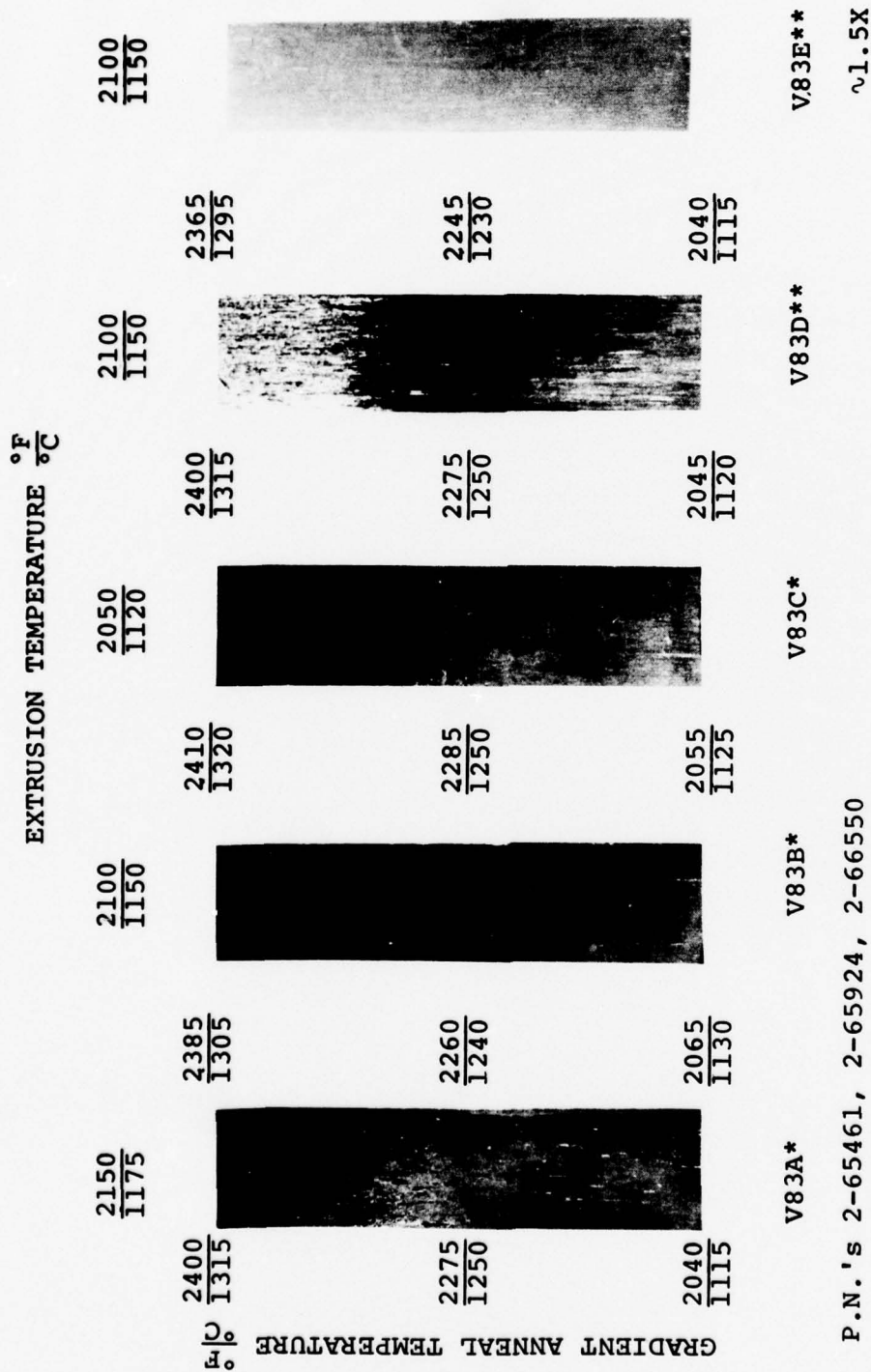


FIGURE 18

MACROGRAPHS OF ALLOY 23 (Ni-15 AT. %Cr-17.5 AT. %Al)  
EXTRUDED (\*100%/\*\*35% PRESS THROTTLE, 18:1 RATIO)  
AND GRADIENT ANNEALED BARS

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>



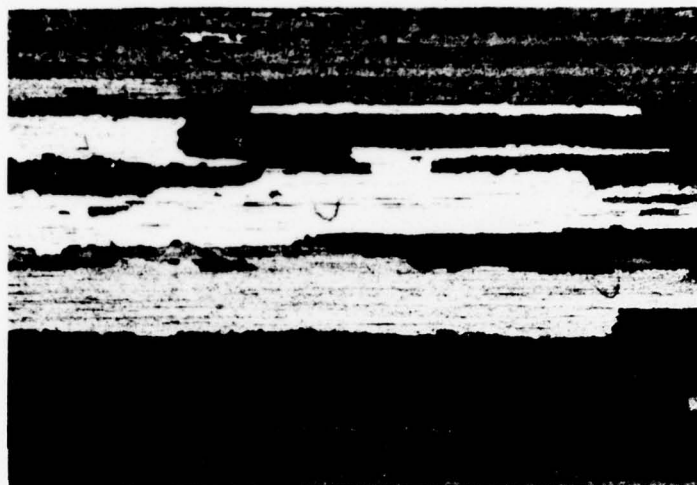
P.N.'s 2-65461, 2-65924, 2-66550

FIGURE 19

MACROGRAPHS OF ALLOY 24 (Ni-20 AT. %Cr-17.5 AT. %Al)  
EXTRUDED (\*100%/\*\*35% PRESS THROTTLE, 18:1 RATIO)  
AND GRADIENT ANNEALED BARS

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>





P.N. 1-52334

20X

FIGURE 20

MICROSTRUCTURE OF ALLOY 2. BAR V21G  
EXTRUDED 2050°F (1120°C)/30:1/4.2 ips  
(10.7 cmph). ZONE ANNEALED 2375°F  
(1300°C)/5.3 iph (13.5 cmph)



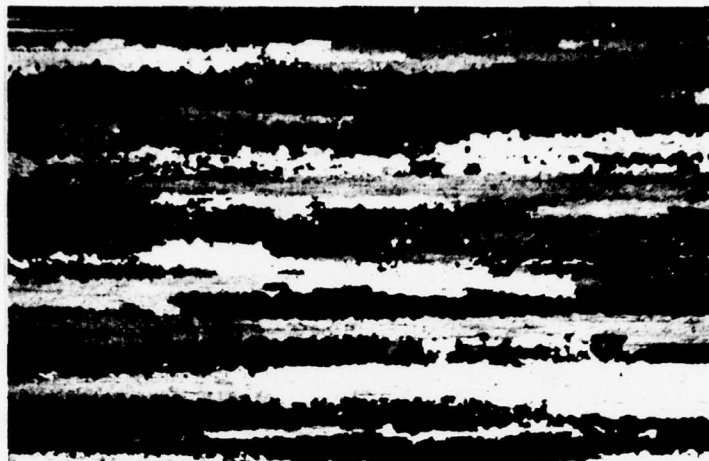
P.N. 1-57891

20X

FIGURE 21

MICROSTRUCTURE OF ALLOY 8. BAR V37F  
EXTRUDED 2050°F (1120°C)/20:1/3.2 ips  
(8.1 cmph)/35% PRESS THROTTLE. ZONE  
ANNEALED 2275°F (1250°C)/5.3 iph  
(13.5 cmph).

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>



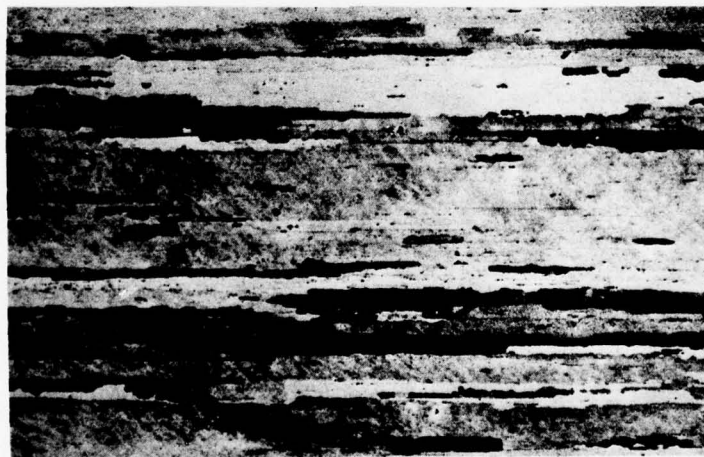
P.N. 1-60569

20X

FIGURE 22

MICROSTRUCTURE OF ALLOY 9. BAR V42A  
EXTRUDED 2100°F(1150°C)/18:1/17.8 ips  
(45.2 cmph). ZONE ANNEALED 2310°F  
(1265°C)/5.3 iph (13.5 cmph).

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>



P.N. 1-61669

20X

FIGURE 23

MICROSTRUCTURE OF ALLOY 12. BAR V49D  
EXTRUDED 2050°F(1120°C)/18:1/11.3 ips  
(28.7 cmph). ZONE ANNEALED 2355°F  
(1290°C)/5.3 iph (13.5 cmph).

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>



P.N. 1-65239

20X

FIGURE 24

MICROSTRUCTURE OF ALLOY 15. BAR V59B  
EXTRUDED 2050°F(1120°C)/18:1/12.1 ips  
(30.6 cmps). ZONE ANNEALED 2245°F  
(1230°C)/3 iph (7.6 cmph).

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>



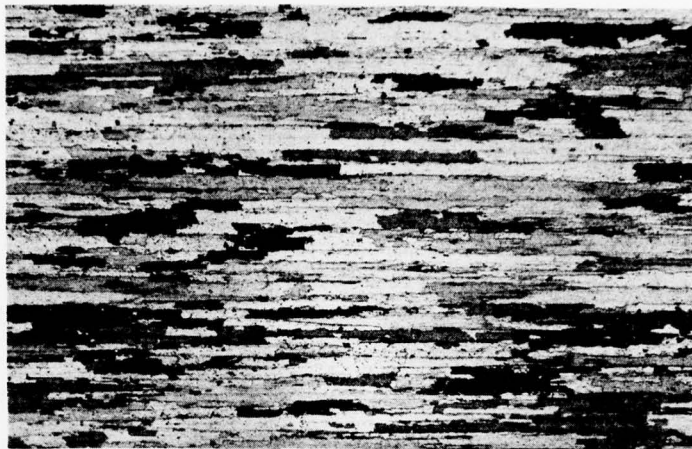
P.N. 1-66540

20X

FIGURE 25

MICROSTRUCTURE OF ALLOY 17. BAR V66D  
EXTRUDED 2100°F(1150°C)/18:1/4.1 ips  
(10.4 cmps). ZONE ANNEALED 2345°F  
(1285°C)/3 iph (7.6 cmph) AND  
ANNEALED 1/2 H/2345°F/AC

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>



P.N. 1-66543

20X

FIGURE 26

MICROSTRUCTURE OF ALLOY 18. BAR V68D  
EXTRUDED 2100°F(1150°C)/18:1/4.2 ips  
(10.7 cmph). ZONE ANNEALED 2350°F  
(1290°C)/3 iph (7.6 cmph) AND  
ANNEALED 1/2 H/2350°F/AC

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>



P.N. 1-65352

20X

FIGURE 27

MICROSTRUCTURE OF ALLOY 19. BAR V71D  
EXTRUDED 2150°F(1175°C)/18:1/35% PRESS  
THROTTLE. ZONE ANNEALED 2350°F(1290°C)/3  
iph (7.6 cmph).

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>





P.N. 1-66546

20X

FIGURE 28

MICROSTRUCTURE OF ALLOY 22. BAR V78D  
EXTRUDED 2100°F(1150°C)/18:1/4.2 ips  
(10.7 cmph). ZONE ANNEALED 2350°F  
(1290°C)/3 iph (7.6 cmph) AND  
ANNEALED 1/2 H/2350°F/AC

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>



P.N. 1-65226

20X

FIGURE 29

MICROSTRUCTURE OF ALLOY 23. BAR V81D  
EXTRUDED 2100°F(1150°C)/18:1/35% PRESS  
THROTTLE. ZONE ANNEALED 2320°F(1270°C)/2.7  
iph (6.8 cmph).

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>

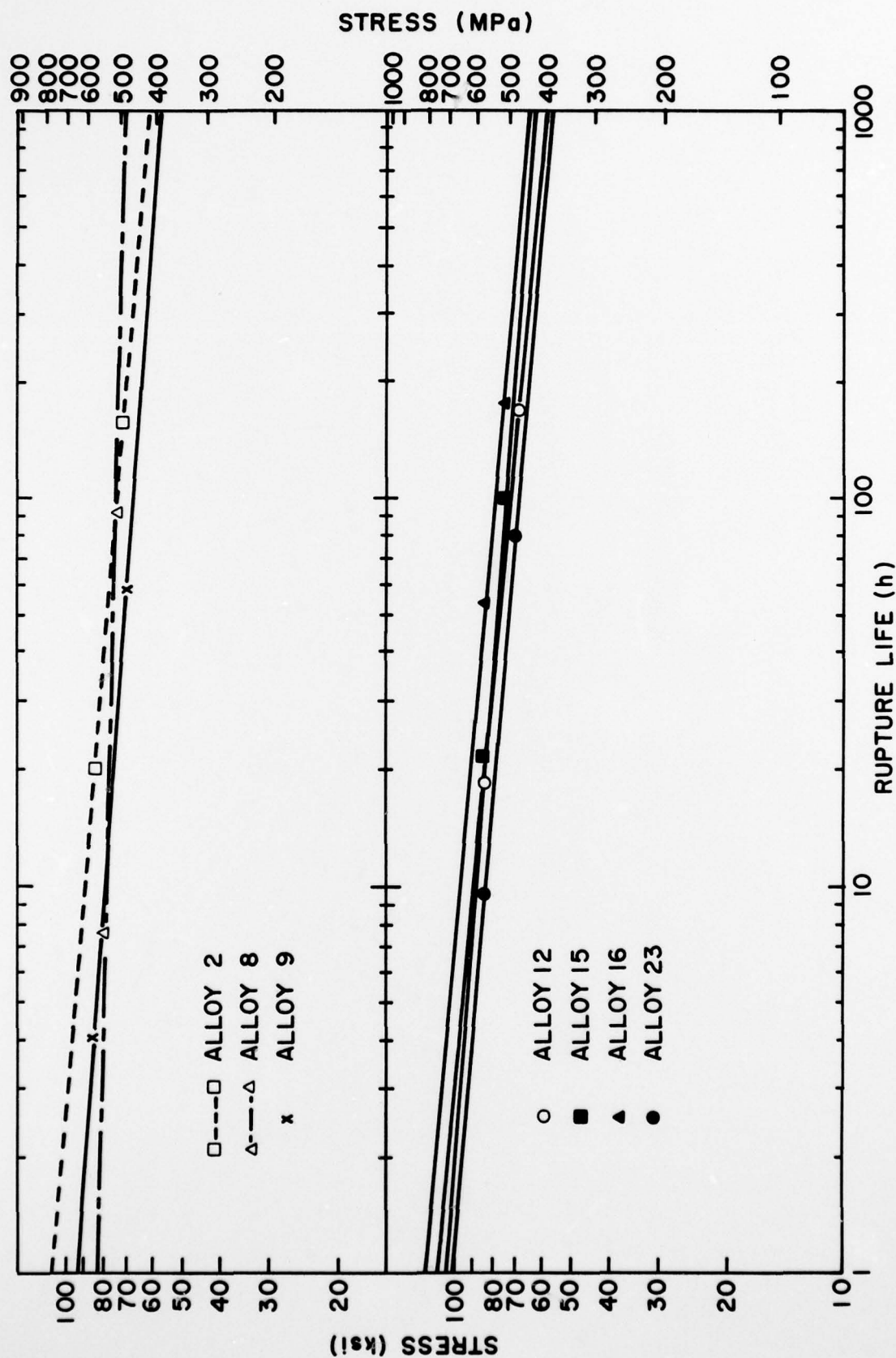


FIGURE 30-1400°F (760°C) STRESS RUPTURE PROPERTIES OF ALLOYS 2, 8, 9, 12, 15, 16 AND 23.

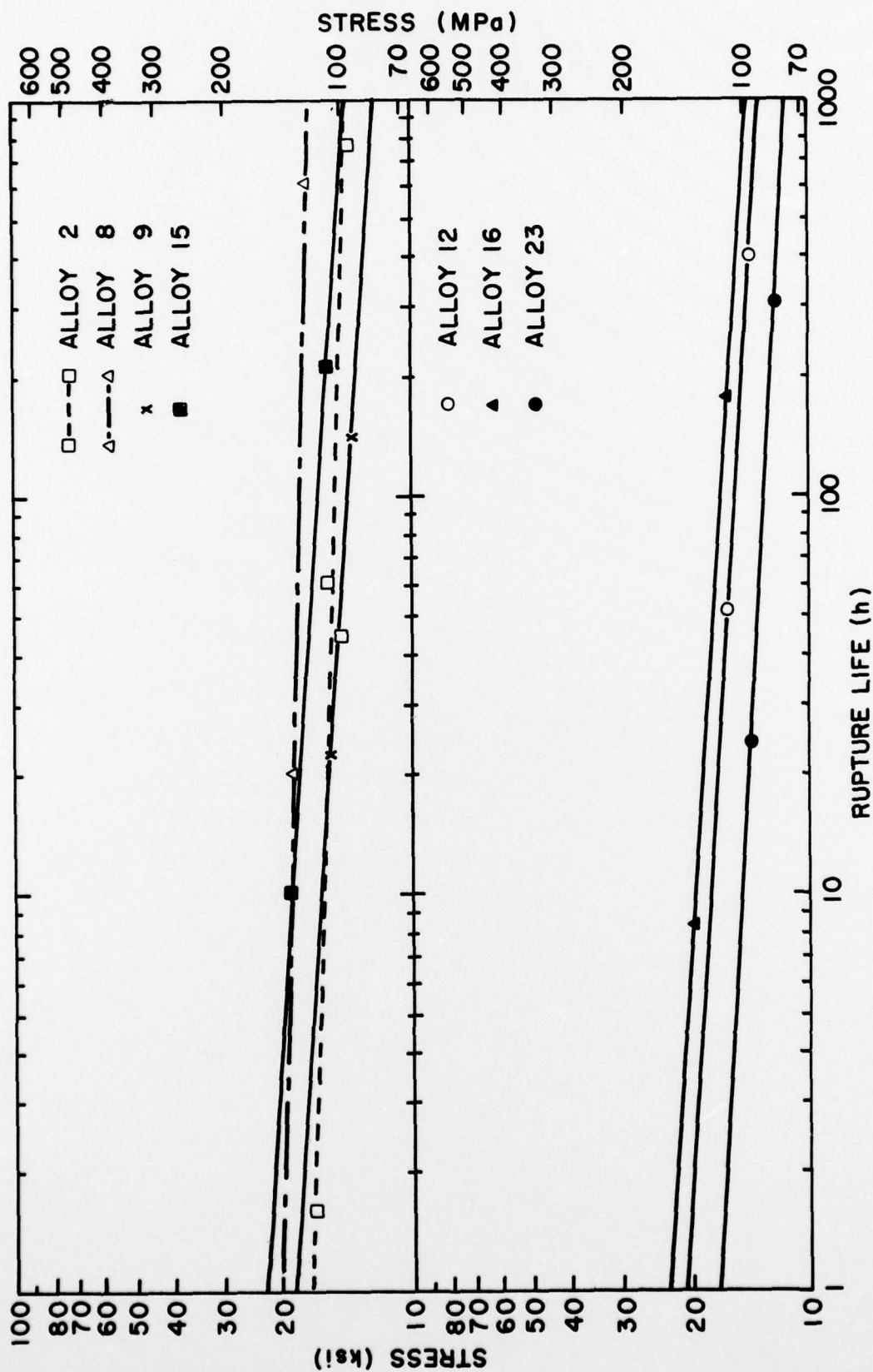


FIGURE 31 - 2000°F (1095°C) STRESS RUPTURE PROPERTIES OF ALLOYS 2, 8, 9, 12, 15, 16 AND 23.

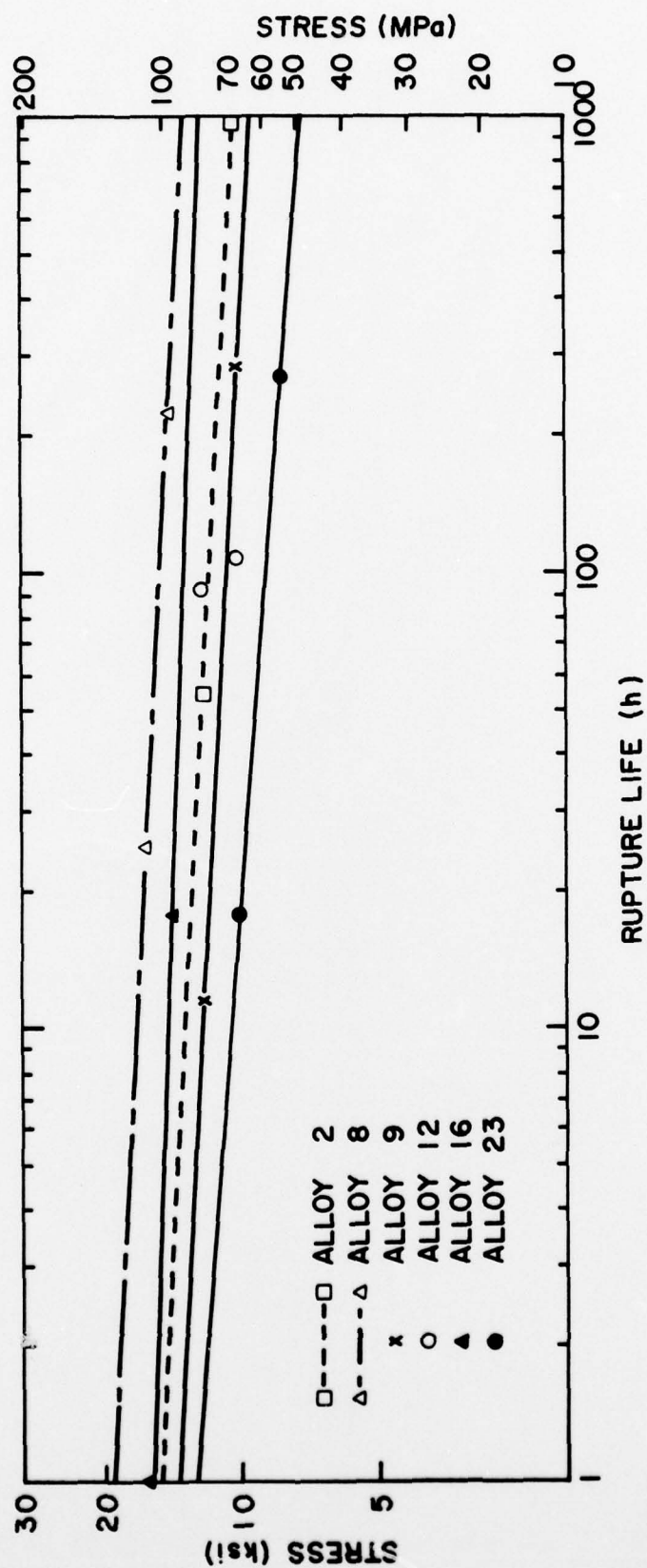
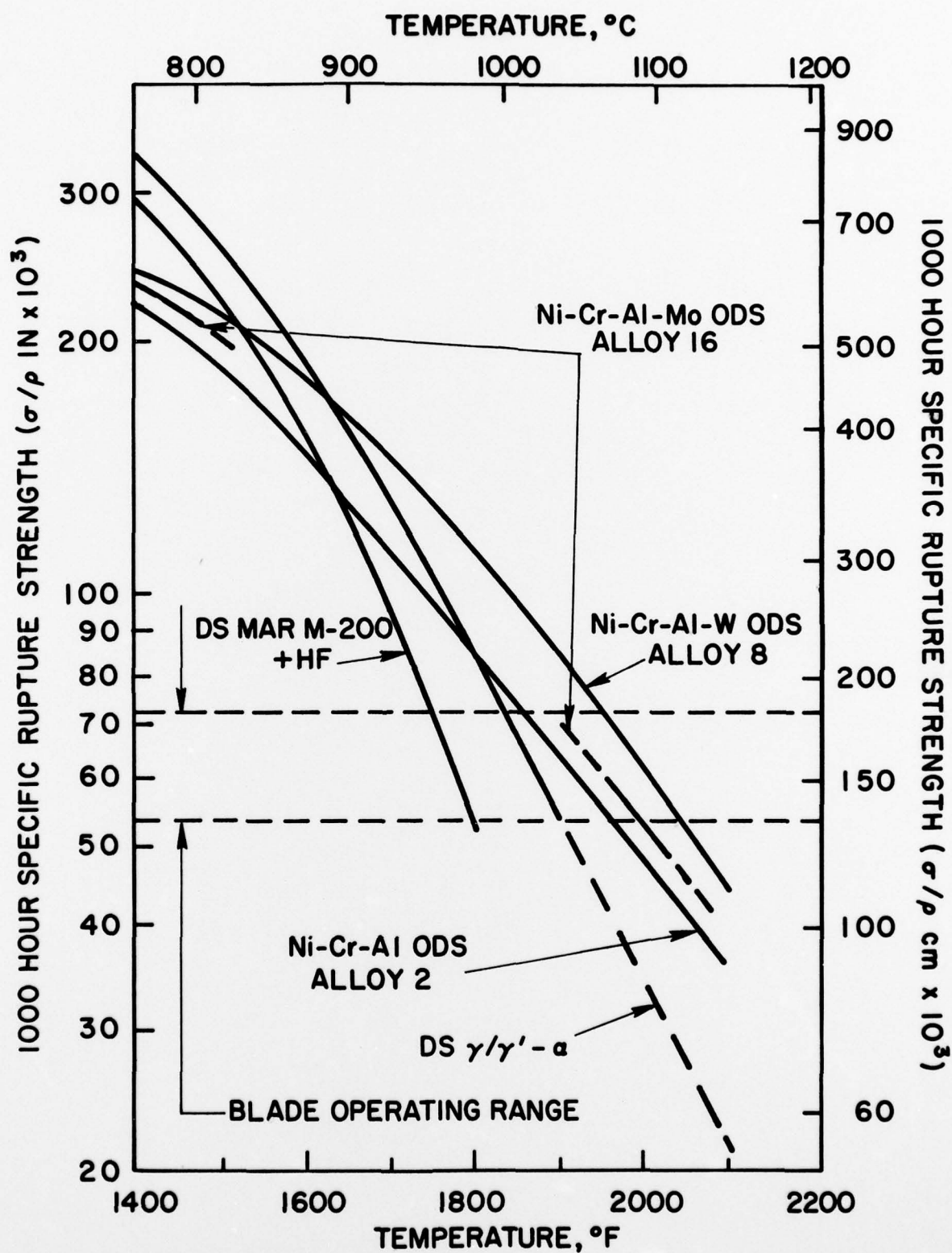
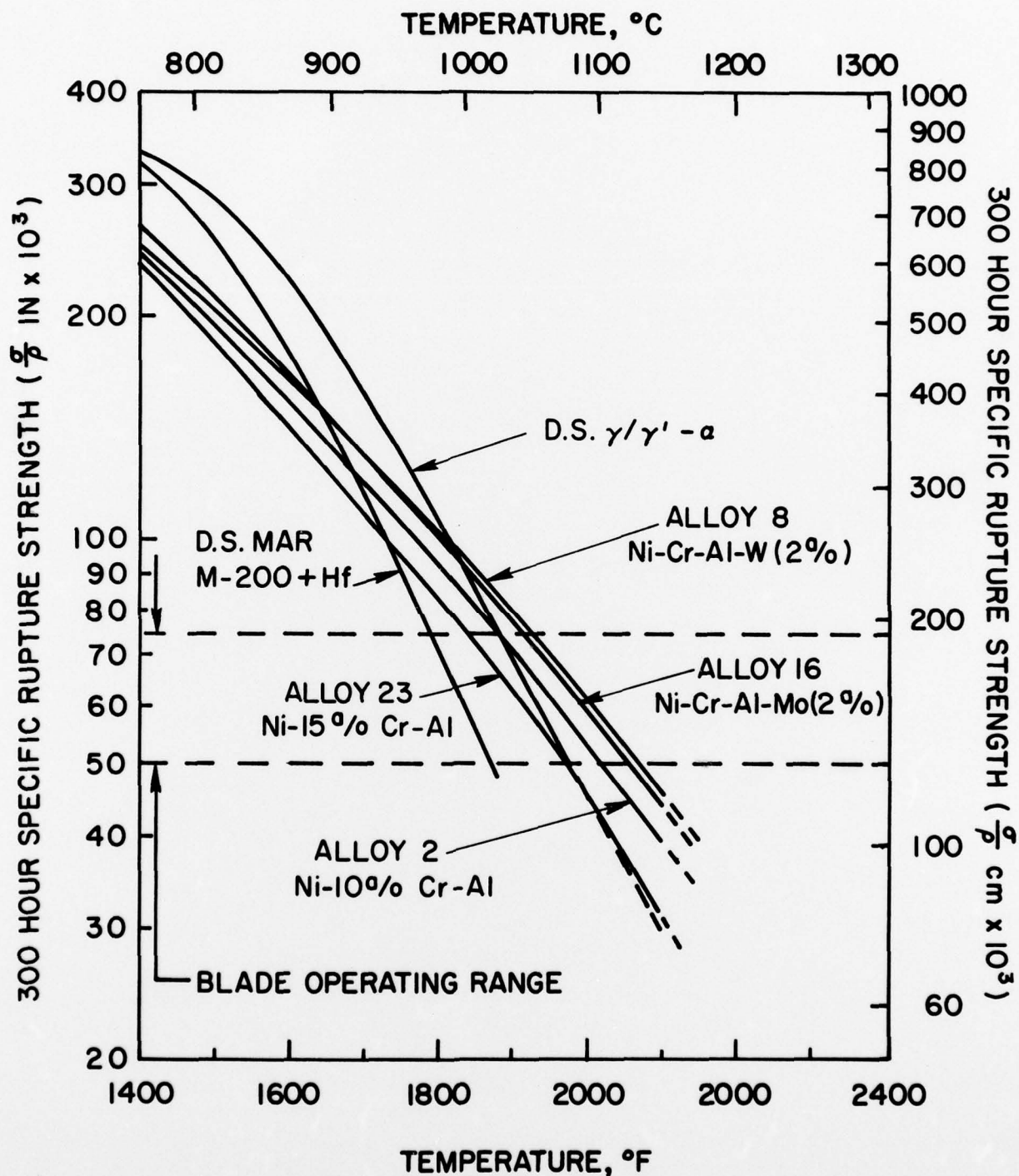


FIGURE 32- 2100°F (1150°C) STRESS RUPTURE PROPERTIES OF ALLOYS 2, 8, 9, 12, 16 AND 23.

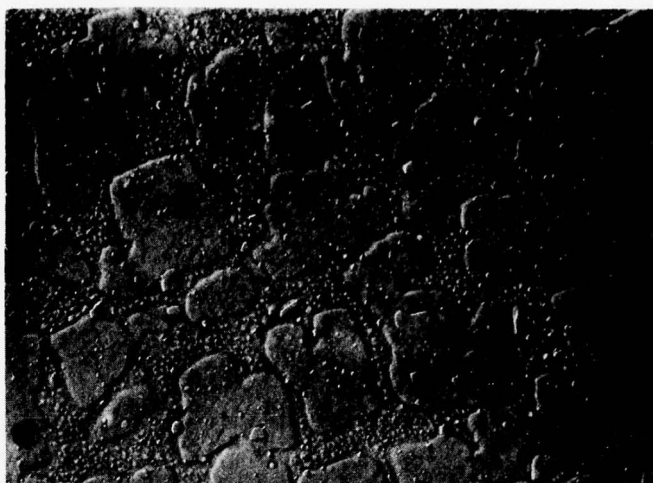




**FIGURE 33-COMPARISON OF 1000 HOUR SPECIFIC RUPTURE STRENGTH TEMPERATURE CAPABILITY OF VARIOUS MATERIALS SYSTEMS.**



**FIGURE 34-COMPARISON OF 300 HOUR SPECIFIC RUPTURE STRENGTH-TEMPERATURE CAPABILITY OF VARIOUS MATERIALS SYSTEMS.**



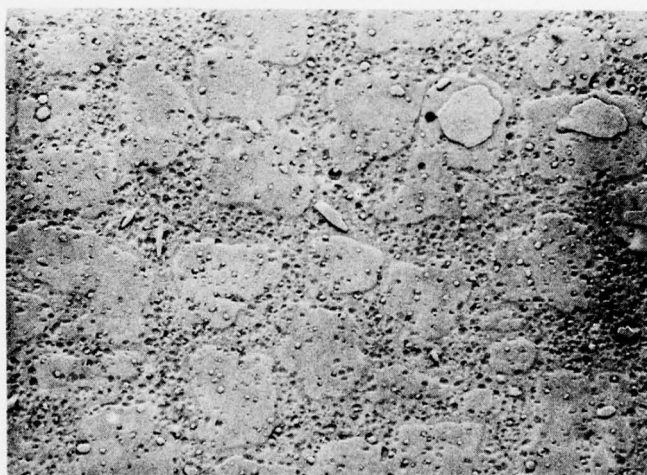
EM-020376

7800x

FIGURE 35

ALLOY 9. BAR V42A, ZONE ANNEALED 2380°F  
(1305°C)/3.0 iph (7.6 cmph). 1/2H/2000°F  
(1095°C)/WQ.  $\gamma/\gamma'$  MORPHOLOGY.

ETCHANT: GLYCEREGIA.



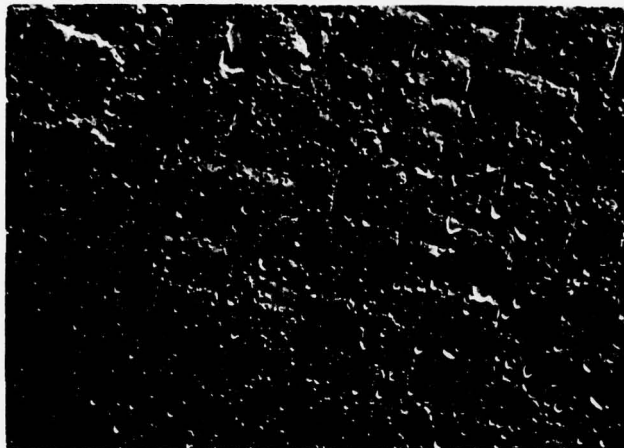
EM-020377

7800X

FIGURE 36

ALLOY 12. BAR V49D, ZONE ANNEALED 2380°F  
(1305°C)/3.0 iph (7.6 cmph). 1/2H/2000°F  
(1095°C)/WQ.  $\gamma/\gamma'$  MORPHOLOGY.

ETCHANT: GLYCEREGIA.



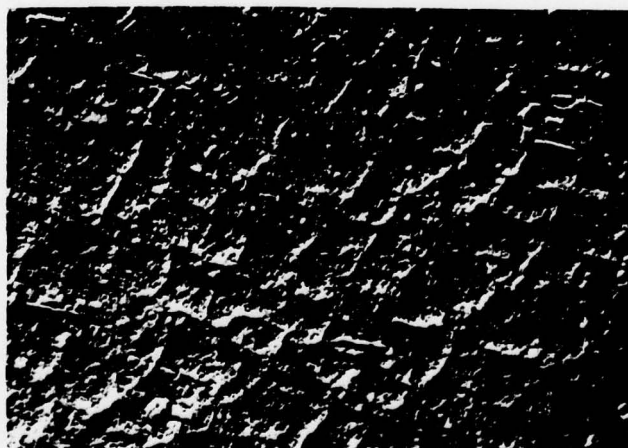
EM-020282

7800X

FIGURE 37

ALLOY 15. BAR V59B, ZONE ANNEALED 2290°F (1255°C)/2.8 iph (7.1 cmph). 1/2H/2000°F (1095°C)/WQ.  $\gamma/\gamma'$  MORPHOLOGY.

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>



EM-020285

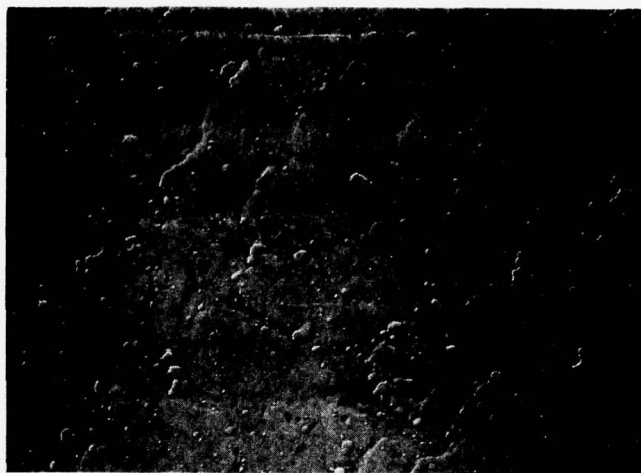
7800X

FIGURE 38

ALLOY 16. BAR V63B, ZONE ANNEALED 2350°F (1290°C)/5.3 iph (13.5 cmph). 1/2H/2000°F (1095°C)/WQ.  $\gamma/\gamma'$  MORPHOLOGY.

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>





EM-020475

7800X

FIGURE 39

ALLOY 8. BAR V37B, ZONE ANNEALED 2330°F  
(1280°C)/2.7 iph (6.9 cmph) + 1/2H/2000°F  
(1095°C)/WQ.  $\gamma/\gamma'$  MORPHOLOGY.



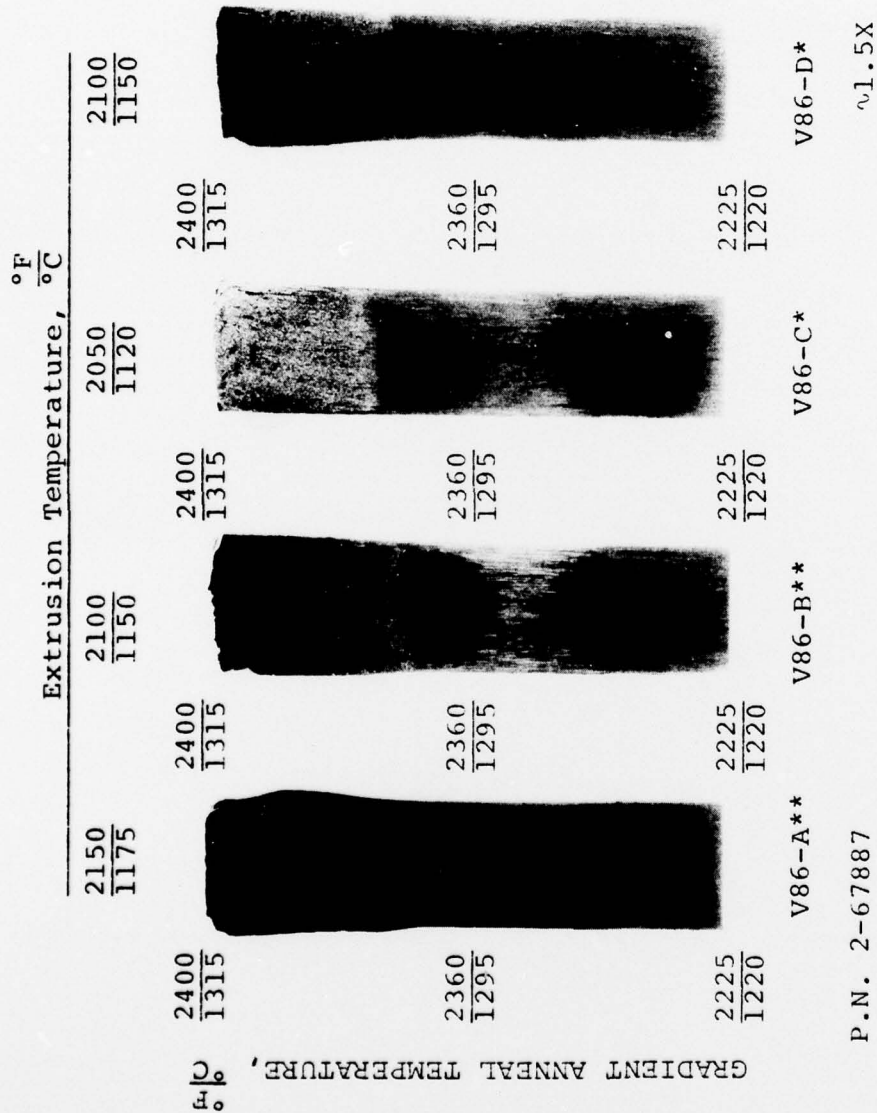
EM-020374

7800X

FIGURE 40

ALLOY 23. BAR V81D, ZONE ANNEALED 2320°F  
(1270°C)/2.7 iph (6.9 cmph) + 1/2H/2320°F  
(1270°C)/AC + 1/2H/2000°F (1095°C)/WQ.  
 $\gamma/\gamma'$  MORPHOLOGY

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>



P.N. 2-67887

FIGURE 41

MACROGRAPHS OF ALLOY 25 (Ni-15AT.%Cr-16.5AT.%Al-10AT.%Co-2AT.%Mo-1AT.%Ta).  
EXTRUDED (\*100%/\*\*35% PRESS THROTTLE, 18:1 RATIO) AND GRADIENT  
ANNEALED BARS

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>

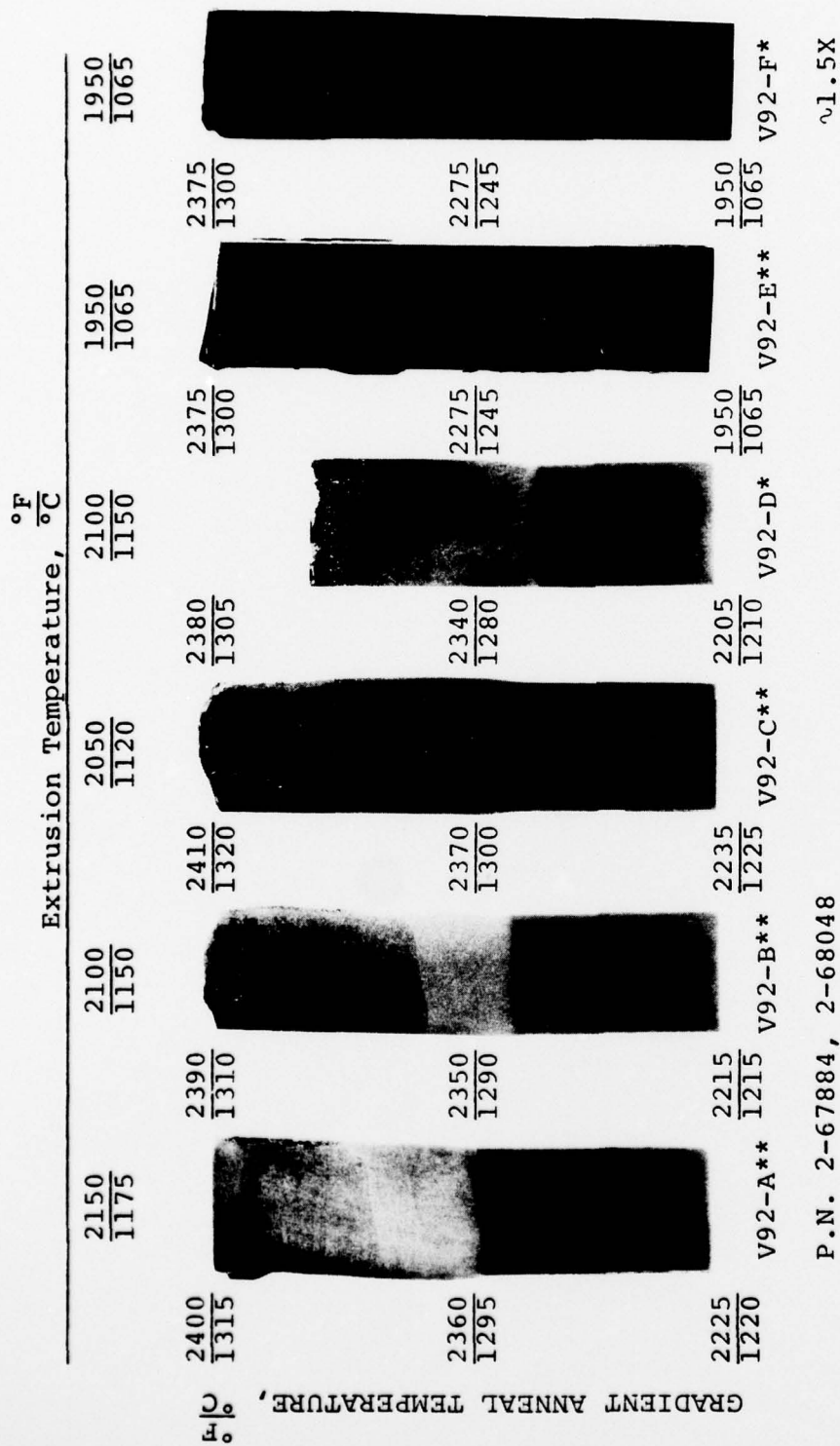


FIGURE 42

MACROGRAPHS OF ALLOY 27 (Ni-15AT.%Cr-16.5AT.%Al-10AT.%Co-2AT.%Mo-2AT.%W-1AT.%Nb).  
EXTRUDED AND GRADIENT ANNEALED BARS.

ETCHANT: 45:45:10, HCl:H<sub>2</sub>O:H<sub>2</sub>O<sub>2</sub>

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